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The Journal of the Society of Dyers and Colourists

Volume 75 Number 8

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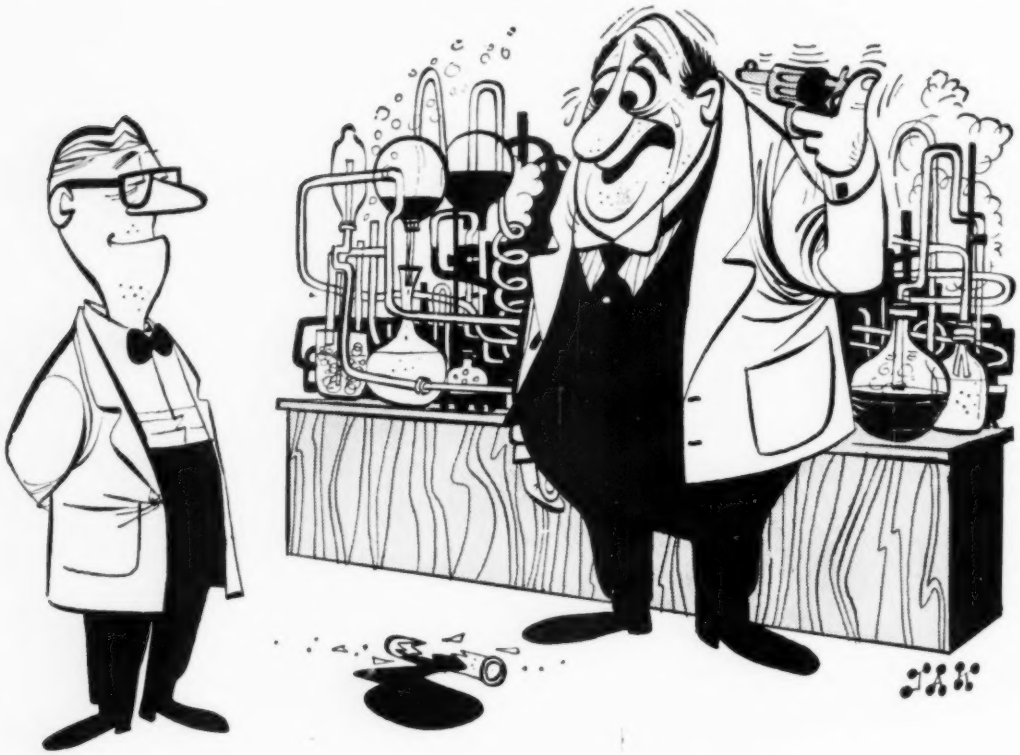

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FORTHCOMING MEETINGS OF THE SOCIETY

Tuesday, 29th September 1959

SCOTTISH SECTION. *Microscopical Studies on Dyeing with the Microdyeoscopes.* H. E. Millson, Esq. (The American Cyanamid Co.). (The lecture will be illustrated with Kodachrome slides and a time-lapse Kodachrome motion picture which includes experiments on the dyeing and treating of wool, rayon, cotton, Dacron, Orlon, Dynel, Creslan and Arnel.) Royal College of Science and Technology, **Glasgow.** 7.15 p.m.

Thursday, 1st October 1959

LONDON SECTION. *Fabrics.* William Brown, Esq., M.Sc. (Joint meeting with the London Centre of the Guild of Dyers and Cleaners). Institute of Journalists, 2/4 Tudor Street, **London E.C.4.** 7.15 p.m.

Friday, 2nd October 1959

LONDON SECTION. *Management.* Prof. R. W. Revans (Department of Industrial Administration, Manchester College of Science and Technology). (Joint meeting with the London Section of the Textile Institute.) Royal Society, Burlington House, **London W.1.** 6 p.m.

Wednesday, 7th October 1959

MIDLANDS SECTION. *Management.* Professor R. W. Revans, B.Sc.(Lond.)., Ph.D.(Camb.). (Joint meeting with the Nottingham Textile Society.) Nottingham and District Technical College, **Nottingham.** 7.15 p.m.

Tuesday, 13th October 1959

NORTHERN IRELAND SECTION. *The Finishing of Fabrics Containing Short Staple 'Terylene'.* D. Clegg, Esq., M.Sc. (Imperial Chemical Industries Ltd.). Grosvenor Rooms, **Belfast.** 7.30 p.m.

Wednesday, 14th October 1959

SCOTTISH JUNIOR BRANCH. *A contribution to the Dyeing of Polyester Fibres.* H. Riding, Esq. (Sandoz Ltd). Technical College, **Paisley.** 7.30 p.m.

Friday, 16th October 1959

WEST RIDING SECTION. Ladies Evening. *Colour and Design in the Home.* Miss Julia Cairns (London Free Lance Journalist for Women's Magazines). Followed by a buffet supper. Victoria Hotel, Bridge Street, **Bradford 1.** 7.30 p.m.

Saturday, 17th October 1959

MIDLANDS SECTION. Ladies Evening. Grand Hotel, **Leicester.** 7 p.m.

Monday, 19th October 1959

BRADFORD JUNIOR BRANCH. *Colour Blindness.* F. Jordinson Esq. (Huddersfield Technical College). Institute of Technology, **Bradford.** 7.15 p.m.

Tuesday, 20th October 1959

SCOTTISH SECTION. *The Choice of an Artificial Daylight for Colour Matching with Special Reference to Fluorescent Materials.* F. L. Warburton, Esq., M.Sc., D.I.C., A.Inst.P. (Wool Industries Research Association). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

HUDDERSFIELD SECTION. *The Development of a new Shrink-resist Finish from the Laboratory to the Mill.* A. N. Davidson, Esq., B.Sc. (W.I.R.A.). (Joint meeting with the Halifax Textile Society.) Silvios Cafe, Westgate, **Huddersfield.** 7.30 p.m.

Thursday, 22nd October 1959

WEST RIDING SECTION. *The Development of a new Shrink-resist Finish from the Laboratory to the Mill.* A. N. Davidson, Esq., B.Sc. (W.I.R.A.). Victoria Hotel, Bridge Street, **Bradford 1.** 7.30 p.m.

MANCHESTER JUNIOR BRANCH. *The Scope for the Junior Technologist in the Textile Industry.* F. M. Stevenson Esq., M.Sc., J.P., The President Elect. Room J/B 16, Chemical Engineering Building (Jackson Street), College of Science and Technology, **Manchester.** 4.30 p.m.

Friday, 23rd October 1959

NORTHERN IRELAND SECTION. Dinner Dance. Woodbourne House Hotel.

LONDON SECTION. The Seventh London Lecture. *Colour and Design in Wallpapers.* Eric A. Entwisle, Esq. (Director, The Wallpaper Manufacturers Ltd.). The Waldorf Hotel, **London W.C.2.** 7 p.m.

Wednesday, 28th October 1959

MIDLANDS SECTION. *Developments in Recent Trends in the Dyeing of Wool.* W. Beal, Esq., B.Sc. King's Head, **Loughborough.** 7 p.m.

Thursday, 29th October 1959

MANCHESTER JUNIOR BRANCH. Cibacron Dyestuffs Exhibition (CIBA Clayton Ltd.). Belle Vue, **Manchester.**

Friday, 6th November 1959

LONDON SECTION. *The Use of Colour in Cosmetics.* D. F. Anstead, Esq., B.Sc., A.R.I.C. Royal Society, Burlington House, **London W.1.** 6 p.m.

WEST RIDING SECTION. ONE DAY CONFERENCE. CREASE RETENTION AND CREASE SHEDDING. *The Permanent Creasing of Wool Fabrics.* Professor J. B. Speakman (Department of Textile Industries, Leeds University). *Crease Retention and Flat Setting of Pure Wool Fabrics.* Dr. A. J. Farnworth (Principal Research Officer, Commonwealth Scientific and Industrial Research Organisation, Australia). *New Reactants and Finishing Agents.* W. A. Bragg, Esq. (Warwick Chemicals (Yorkshire) Ltd.).*The Crease recovery of Wet Fabrics.* Dr. W. Lonie (British Rayon Research Association). Leeds University, **Leeds 2.** 2 p.m.

Monday, 9th November 1959

HUDDERSFIELD SECTION. *The Dyeing of Man-made Fibres.* H. Birchall, Esq. (Wm. North & Sons Ltd). Invitation from the Halifax Textile Society. Joint meeting at The Alexandra Hall, **Halifax.** 7.30 p.m.

Tuesday, 10th November 1959

NORTHERN IRELAND SECTION. *The Theory and Practice of Sodium Chlorite Bleaching.* J. K. Skelly, Esq., M.Sc., Ph.D., A.R.I.C., F.S.D.C. (College of Technology, Belfast). Grosvenor Rooms, **Belfast.** 7.30 p.m.

Thursday, 12th November 1959

MANCHESTER JUNIOR BRANCH. *A New Mechanism of Detergency.* A. S. C. Lawrence Esq., Ph.D., Sc.D., F.R.I.C., F.Inst.Pet. Room J/B 16, Chemical Engineering Building (Jackson Street), College of Science and Technology, **Manchester.** 4.30 p.m.

SCOTTISH JUNIOR BRANCH. *The Development of a New Shrink-resist Finish from the Laboratory to the Mill.* A. N. Davidson, Esq., B.Sc. (Wool Industries Research Association). Technical College, **Paisley.** 7.30 p.m.

WEST RIDING SECTION. *An investigation into the Application of Logwood to Dicel and Nylon Fabrics.* E. France, Esq. (Courtaulds Ltd., Bocking). Victoria Hotel, Bridge Street, **Bradford 1.** 7.30 p.m.

Tuesday, 17th November 1959

SCOTTISH SECTION. *The Finishing of Fabrics containing Short Staple Terylene.* D. Clegg, Esq., M.Sc., A.T.I. (Imperial Chemical Industries Ltd.). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

HUDDERSFIELD SECTION. *Recent Developments in Wool Dyeing.* D. G. Evans, Esq., B.Sc. (CIBA Clayton Ltd.). Silvios Cafe, Westgate, **Huddersfield.** 7.30 p.m.

Wednesday, 18th November 1959

MIDLANDS SECTION. *Identification of Dispersed Dyes.* W. L. Lead, Esq., Ph.D., F.R.I.C., D. Haig, Esq., F.T.I., F.S.D.C., L. A. Telesz, Esq., F.T.I. College of Technology, **Leicester.** 7 p.m.

Thursday, 26th November 1959

WEST RIDING SECTION. *The Principles of Colour Photography.* D. P. Ayres, Esq., B.Sc. (Ilford Ltd.). Griffin Hotel, Boar Lane, **Leeds 1.** 7.30 p.m.

Friday, 4th December 1959

LONDON SECTION. Short Papers by London Section Members. *Dyeing of Ribbons.* R. C. Oakley, Esq. and *Dyeing of Carpet Yarns.* R. Woods, Esq., F.S.D.C. Royal Society, Burlington House, **London W.1.** 6 p.m.

Tuesday, 8th December 1959

SCOTTISH JUNIOR BRANCH. *Heat Setting.* J. A. Bamber, Esq. (British Rayon Research Association). Technical College, **Paisley.** 7.30 p.m.

Thursday, 10th December 1959

WEST RIDING SECTION. *The Kinetics of Wool Dyeing with 2,1 Metal Complexes.* Dr. H. Ackermann (Geigy, Basle). Victoria Hotel, Bridge Street, **Bradford 1.** 7.30 p.m.

Tuesday, 15th December 1959

HUDDERSFIELD SECTION. *The Colouring of Tufted Carpet in Piece Form.* R. C. Cheetham Esq., G. W. Lewis, Esq. and Dr. J. Waller. (Courtaulds Ltd., Droylsden). Silvios Cafe, Westgate, **Huddersfield.** 7.30 p.m.

SCOTTISH SECTION. *The Principles of Colour Photography.* Dr. C. H. Giles (on behalf of Ilford Ltd.). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

continued on page iv

FORTHCOMING MEETINGS OF THE SOCIETY — *continued from page* iii

Tuesday, 15th December 1959

NORTHERN IRELAND SECTION. *The Mechanism of Dyeing with Procion Dyes.* H. H. Sumner, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Grosvenor Rooms, **Belfast.** 7.30 p.m.

Wednesday, 16th December 1959

MIDLANDS SECTION. *Acrilics in Knitwear.* Half Day Symposium. J. F. Gaunt, Esq., Ph.D., F.R.I.C., F.T.I., H. D. Edwards, Esq., Ph.D., A.R.I.C., J. Rayment, Esq., A.T.I. College of Technology, **Loughborough.** 2.30 p.m. and 7 p.m.

Friday, 8th January 1960

LONDON SECTION. *New Works on the Dyeing of Nylon with Disperse Dyes.* H. W. Seaman, Esq., B.A. (I.C.I. Ltd.). Royal Society, Burlington House, **London W.1.** 6 p.m.

Tuesday, 12th January 1960

NORTHERN IRELAND SECTION. *Radio-isotopes and Their Industrial Applications.* R. J. Magee, Esq., M.Sc., Ph.D., M.R.S.A., A.R.I.C. (Queen's University, Belfast). Grosvenor Rooms, **Belfast.** 7.30 p.m.

Thursday, 14th January 1960

WEST RIDING SECTION. *Experience gained in the Dyeing and Finishing of Polyester/Wool Fabrics in Germany.* Dr. J. Nusslein (Farbwerke Hoechst). Victoria Hotel, Bridge Street, **Bradford 1.** 7.30 p.m.

Tuesday, 19th January 1960

HUDDERSFIELD SECTION. (Title to be announced later.) Dr. J. Nusslein (Farbwerke Hoechst A.G.). Silvios Cafe, Westgate, **Huddersfield.** 7.30 p.m.

MIDLANDS SECTION. *Beam Dyeing.* (Paper written by Burlington Corp. U.S.A.) K. S. Laurie, Esq. A.M.I.Mech.E., A.M.I.E.E. Gas Theatre, **Nottingham.** 7 p.m.

SCOTTISH SECTION. *Some Problems on the Dyeing and Finishing of Fabrics using the Newer Synthetics from the point of view of the customer.* J. S. Ingham, Esq., M.Sc., F.R.I.C. (Marks & Spencer Ltd.). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Friday, 22nd January 1960

HUDDERSFIELD SECTION. Annual Dinner. Princess Cafe, **Huddersfield.**

WEST RIDING SECTION. Annual Dinner and Dance.

Thursday, 28th January 1960

WEST RIDING SECTION. *Levelling Agents for Wool Dyeing.* Dr. J. Lanter (CIBA, Basle). Victoria Hotel, Bridge Street, **Bradford 1.** 7.30 p.m.

Monday, 1st February 1960

HUDDERSFIELD SECTION. *Choice of an Artificial Daylight for Colour Matching with Special Reference to Fluorescent Materials.* F. L. Warburton, Esq., M.Sc., A.R.C.S., D.I.C., A.Inst.P. (Wool Industries Research Association). (Joint meeting with Huddersfield Textile Society.) Large Hall, College of Technology, **Huddersfield.** 7.30 p.m.

Tuesday, 2nd February 1960

NORTHERN IRELAND SECTION. *Professional Salesmanship—The Key to Prosperity.* A. B. M. Taylor, Esq. (Irish Linen Guild). (Joint meeting with Textile Institute.) York Street Flax Spinning Co. Ltd., Henry Street, **Belfast.** 7.30 p.m.

Friday, 5th February 1960

LONDON SECTION. *Dyeing and Finishing of 'Courtolon' Nylon in relation to its Yarn Properties.* L. Cotton, Esq., B.A., and J. Wardle, Esq. (Courtaulds Ltd.). Royal Society, Burlington House, **London W.1.** 6 p.m.

Tuesday, 9th February 1960

NORTHERN IRELAND SECTION. *Problems Relating to Permanent Finishes.* Dr. A. Schurch (CIBA Ltd., Basle). Grosvenor Rooms, **Belfast.** 7.30 p.m.

Wednesday, 10th February 1960

MIDLANDS SECTION. *Dyeing and Finishing of Courtolon Yarns, Fabrics and Garments.* J. Wardle, Esq., L. Cotton, Esq., B.Sc. College of Technology, **Leicester.** 7 p.m.

Thursday, 11th February 1960

WEST RIDING SECTION. *Paper Chromatography of Dyestuffs.* J. C. Brown, Esq. (CIBA Clayton Ltd.). Griffin Hotel, Boar Lane, **Leeds 1.** 7.30 p.m.

SCOTTISH SECTION *Recent Developments in Wool Dyeing.* W. Beal, Esq., B.Sc. (Geigy Co. Ltd.). (By invitation of the Textile Institute.) Scottish Woollen Technical College, **Galashiels.** 7.30 p.m.

Friday, 12th February 1960

LONDON SECTION. Annual Dinner and Dance. Waldorf Hotel, **London W.C.2.** 7 p.m. for 7.30 p.m.

Tuesday, 16th February 1960

SCOTTISH SECTION. *The Keratin of Wool.* Professor J. B. Speakman, D.Sc., F.R.I.C., F.T.I. (Joint meeting with the Textile Institute.) The Royal College of Science and Technology, **Glasgow.** 7.30 p.m.

Tuesday, 23rd February 1960

SCOTTISH SECTION. *Some observations on the Production of Dyed Yarn for Carpets.* G. B. Angus, Esq., B.Sc., A.R.I.C., A.M.I.I.A., F.S.D.C. (J. Crossley & Sons). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Thursday, 25th February 1960

WEST RIDING SECTION. *New Work on the Dyeing of Nylon with Disperse Dyes.* H. Seaman, Esq., (I.C.I. Ltd.). Victoria Hotel, Bridge Street, **Bradford 1.** 7.30 p.m.

Friday, 4th March 1960

LONDON SECTION. *Recent Developments in Leather Dyeing.* J. H. Sharphouse, Esq. (Northampton College of Technology). Royal Society, Burlington House, **London W.1.** 6 p.m.

Tuesday, 8th March 1960

NORTHERN IRELAND SECTION. *The Dyeing and Finishing of Fabrics Containing Tricel.* R. J. Mann, Esq., B.Sc., F.S.D.C. (Courtaulds Ltd.). Grosvenor Rooms, **Belfast.** 7.30 p.m.

HUDDERSFIELD SECTION. *Paper Chromatography of Dyestuffs.* J. C. Brown, Esq., B.Sc., A.R.I.C. (CIBA Clayton Ltd.). (Joint meeting with the Huddersfield Section of the Royal Institute of Chemistry.) Silvios Cafe, Westgate, **Huddersfield.** 7.30 p.m.

Thursday, 10th March 1960

MIDLANDS SECTION. *The Dyeing of Ribbons.* E. Bridge, Esq. (Joint meeting with the Coventry Textile Society.) Courtaulds Acetate and Synthetic Fibres Laboratory, **Coventry.** 7.30 p.m.

WEST RIDING SECTION. *A comparison of the Dyeing of Acrylics and Worsted Hosiery Yarns.* Dr. J. F. Gaunt (Patons and Baldwins Ltd.). Victoria Hotel, Bridge Street, **Bradford 1.** 7.30 p.m.

Tuesday, 15th March 1960

SCOTTISH SECTION. Annual General Meeting 7 p.m. Followed by *Paper Chromatography of Dyestuffs.* J. C. Brown, Esq. (CIBA Clayton Ltd.) St. Enoch Hotel, **Glasgow.** 7.30 p.m.

Wednesday, 16th March 1960

MIDLANDS SECTION. *Thermal Setting of Tricel.* W. F. Kilby, Esq., B.Sc., A.Inst.P. (Joint meeting with B.A.O.C.) Midland Hotel, **Derby.** 7 p.m.

Friday, 18th March 1960

MIDLANDS SECTION. Annual Dinner. George Hotel, **Nottingham.** 7 p.m.

Thursday, 24th March 1960

WEST RIDING SECTION. Annual General Meeting followed by *Problems involved in Piece Dyeing with Vat Dyestuffs.* M. Brauer, Esq. (Badische Anilin & Soda Fabrik A.G.). Victoria Hotel, Bridge Street, **Bradford 1.** 7.30 p.m.

Friday, 25th March 1960

LONDON SECTION. *The Origin of Faults in the Dyeing of Textiles.* N. Jackson, Esq. (Courtaulds Ltd.). George Hotel, **Luton.** 7 p.m.

Thursday, 7th April 1960

MIDLANDS SECTION. *Recent Developments in Dyeing Wool.* D. B. Evans, Esq. (Joint meeting with the Textile Institute.) Carpet Trades Ltd., Canteen, **Kidderminster.** 7.30 p.m.

Tuesday, 12th April 1960

SCOTTISH SECTION. *Ladies Evening.* (Details to be announced later.)

Wednesday, 13th April 1960

MIDLANDS SECTION. Annual General Meeting. *Colour Photography.* D. P. Ayres, Esq., B.Sc., A.R.P.S. College of Technology, **Leicester.** 7 p.m.

Tuesday, 19th April 1960

HUDDERSFIELD SECTION. Annual General Meeting. Followed by a lecture *How Colour Photography Works.* D. P. Ayres, Esq., B.Sc. (Ilford Ltd.). Silvios Cafe, Westgate, **Huddersfield.** 7.30 p.m.

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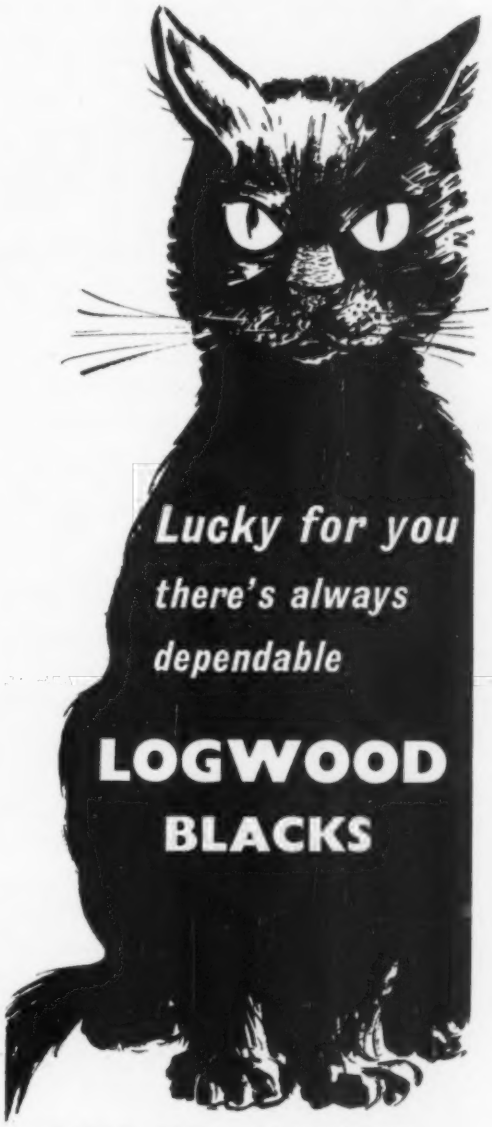

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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc, should consult pages 1–10 of the January 1959 and pages 341–348 of the July 1959 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138). *Editorial Communications* should be addressed to *The Editor*, at the same address

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

New Ideas in the Application of Phthalocyanine Derivatives to Textiles — *E. Gund*

Some Recent Advances in Textile Printing — *R. J. Hannay*

Some Relations between the Chemical Constitution of Cibacron Dyes and their Dyeing Characteristics — *J. Wegmann*

COMMUNICATIONS

Studies on Haematoxylin and Haematein, the Colouring Principles of Logwood

I— Absorption Spectra of Pure Compounds in Various Solvents and a Spectrophotometric Method of Analysis for Haematoxylin and Haematein

II— Behaviour in Aqueous Solution at Varying pH and p*K* Values

G. C. Lalor and S. L. Martin

The Separation, Estimation, Orientation, and Ultraviolet Spectra of Isomeric Azo Compounds formed by Coupling to 1-Naphthylamine and its Derivatives

E. R. Ward, B. D. Pearson, and P. R. Wells

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THE JOURNAL

OF THE

Society of Dyers and Colourists

Volume 75 Number 8 AUGUST 1959* Issued Monthly

Proceedings of the Society

Emulsion Thickenings—Possibilities in Textile Printing

T. L. DAWSON

Meetings of the Northern Ireland Section held in the Grosvenor Rooms, Belfast, on 13th January 1959, Mr. W. J. Macnab in the chair; and of the Manchester Section held at the Textile Institute, Manchester, on 20th February 1959, Mr. J. W. Reidy in the chair

The use of emulsion thickenings for printing dyes, other than pigment compositions, on both natural and synthetic fibres is discussed. Theoretical and practical aspects of these emulsion systems are considered, and the advantages of oil-in-water emulsions over the water-in-oil type are set out. The rheological properties of emulsion thickenings are compared with those of some conventional thickenings, and the practical implications of these results are discussed. Details of recipes for the preparation of stock emulsion thickenings for the application of a wide range of dyes (including reactive, vat, onium, and azoic dyes) are given, and the technical advantages resulting from their use enumerated.

1. INTRODUCTION

In recent years, particularly on the Continent and in the U.S.A., increasing attention has been paid to the possibilities of printing dyes, other than pigment printing compositions, from emulsion-thickened print pastes. Before the practical application and the advantages of using such systems are discussed, it is necessary to appreciate the underlying principles from both the theoretical and the practical standpoint.

(*a*) *Theoretical Considerations*

Emulsions may be of the water-in-oil or the oil-in-water type, in which the first- and second-named phases are the disperse and the continuous phases respectively. Stable emulsions are usually formed only in the presence of a third component, the emulsifying agent. The type of emulsion that is formed under given conditions was originally thought to depend upon the phase volume relationship: usually, if the oil phase amounts to 5–40% by volume of the emulsion, a water-in-oil emulsion is formed; whereas if the proportion is 40–75%, an oil-in-water emulsion is formed. This is only partially true, and nowadays the polar–non-polar balance of the emulsifying agent is regarded as being of greater importance. For example, alkali-metal soaps favour the formation of oil-in-water emulsions, whereas the alkaline-earth metal soaps (which are less hydrophilic than the alkali-metal soaps) favour the formation of water-in-oil emulsions. Theoretically an emulsion containing more than 74% by volume of the disperse phase is not possible, as this represents the closest packing of spheres. This assumes, however, that the droplets of the disperse phase are all of equal diameter and cannot be distorted. In practice, emulsions containing considerably larger volumes of disperse phase can be prepared.

For an emulsion to be stable the interfacial tension between the two phases must be low; otherwise the globules would tend to coalesce and bring about a reduction in the free energy of the system. The emulsifier is thought to form a layer between the disperse and continuous phases, which acts as a sort of diaphragm. This diaphragm will have a definite interfacial tension against the oil on one side and against the water on the other side. If the tension between the emulsifier layer and the oil phase is less than that between the emulsifier layer and the aqueous phase, the oil phase will tend to form a convex surface and hence lead to an oil-in-water emulsion. With the reverse order of tensions a water-in-oil emulsion results. The relative magnitude of the tensions in the system will, in turn, depend upon the relative dispersibility of the emulsifier in the two phases.

Apart from a few emulsifiers of the lyophilic colloid type (e.g. casein and gum senegal) and the finely divided insoluble emulsifiers (e.g. carbon black and bentonite), most compounds used to prepare emulsions are hydrophobic organic compounds with polar groups attached. The type of emulsion that these emulsifiers favour depends upon the ratio of polar to non-polar groups present.

The dispersed oil particles in an oil-in-water emulsion tend to carry a charge at their surface, the polarity of which depends on the emulsifying agent used. It is reasonable to suppose that these

* Published September 1959

charges (their polarity is immaterial) assist in stabilising the emulsion, and any additions to the emulsion which tend to remove the charge (e.g. multivalent ions of opposite charge) will result in a reduction in stability.

To distinguish between oil-in-water and water-in-oil emulsions two simple tests may be applied. If mixed with water, an oil-in-water emulsion remains stable and visually homogeneous, while a water-in-oil emulsion breaks and does not mix. An oil-in-water emulsion can also conduct electricity, whereas a water-in-oil emulsion cannot, since oil is the continuous phase.

(*b*) *Practical Considerations*

For printing applications the emulsion thickening must be stable (*a*) to the dye used and (*b*) to the other constituents of the print paste (e.g. salt, acid, alkali, and metal salts). Thus anionic dyes cannot in general be used with cationic dispersing agents, and *vice versa*. In a similar manner additions of electrolyte can e.g. affect either the distribution of the emulsifier between the aqueous and the oil phases or the charge on the particles of the disperse phase, so that the emulsion tends to *invert* (change its type) or to *break* (separate into two layers). Some emulsifying agents, although compatible with the dye, may affect its fixation. Thus Lissolamine A (ICI), for example, cannot be used in conjunction with vat or onium dyes because of its restraining action.

The viscosity of the emulsion must be within a suitable range for application by printing (say, 500–5,000 centipoises, depending on the method of application and the fabric being printed), and this is controlled by adjustment of the phase volume relationship (i.e. the proportion of disperse phase). Addition of water to an oil-in-water emulsion reduces the viscosity, while oil-phase additions cause the same change in water-in-oil emulsions. For this reason oil-in-water emulsions are more easily washed off from printing rollers than water-in-oil emulsions, the latter tending to thicken and finally break on admixture with excess water, while the former become thin and remain emulsified. When the emulsion breaks, the oil phase collects on the surface of the aqueous phase, and this introduces a fire hazard. Oil-in-water emulsions are usually less difficult to prepare than water-in-oil types and tend to be more stable towards electrolyte additions. Low-speed stirrers are often sufficient to produce oil-in-water emulsions, whereas high-speed stirring and homogenisation are usually required for the production of the water-in-oil type. If, as in normal colour shop practice, a stock thickening is prepared, the oil-in-water emulsions are made more amenable to subsequent additions of aqueous dye pastes or solutions, since these additions require no further emulsification, as would be the case with water-in-oil stock thickenings.

Water-in-oil emulsions are reported[1] to have superior lubricating properties to oil-in-water emulsions, which in turn are superior to conventional thickenings. This effect is presumably due to the lubricating action of the oil phase, this being greatest when the oil is in the external phase. The superior properties of emulsion thickenings result in a reduced tendency for roller and doctor blade damage, which cause streaking or scumming in printing.

For these reasons oil-in-water emulsions are preferable technically. On the other hand, water-in-oil emulsions are economically more attractive, because of the lower consumption of hydrocarbon oil.

Both types of emulsion exhibit pseudo-plastic flow (see p. 416), water-in-oil emulsions more so than the oil-in-water type. Thus under shearing action, such as exists between machine printing rollers and doctor blades, the emulsion flows, but once it has been applied to the fabric, thickening occurs, and no further flow results. Hence machine-printed emulsions tend to give, for example, unlevel blotch prints. This defect can be overcome to some extent by crushing, and also by the addition of an auxiliary film-forming agent having good flow properties, e.g. sodium alginate. Such additions also— (i) assist in preventing the spread of the aqueous phase (and dye) beyond the edges of the printed areas by capillary attraction; (ii) may act as protective colloids and thus stabilise the emulsion; and (iii) contribute to the viscosity of the emulsion, since this latter property depends, to some extent, on the viscosity of the aqueous phase. The spread of the aqueous phase over the fabric is least if the surface-active agent chosen, while giving a low interfacial tension between oil and aqueous phases, does not give too large a reduction of the surface tension of the aqueous phase relative to that of pure water.

2. EXPERIMENTAL

(*a*) *Materials*

Dyes— These were all of commercial quality and comprised mainly dyes from the Procion, Caledon, Durindone, Alcian X, and Brentogen (ICI) ranges.

Film-forming Agents— The following commercially available thickening agents were used as film formers in the emulsion systems described—

Manutex RS Manutex C Manutex SX/LD	Alginate Industries Ltd., London
Celacol MM 10 Celacol M 2500	British Celanese Ltd., London
Solvitose C 5	W. A. Scholten's Chemische Fabrieken NV., Groningen, Holland
Meypro Gum CRX	Meypro Ltd., Weinfelden, Switzerland
Indalca U	Cesalpinia, Milan, Italy
Gum tragacanth	James Laing, Son & Co., Manchester

(*b*) *Surface Tension Measurements*

A micrometer Agla micropipette fitted with a hypodermic needle, the end of which had been ground flat, was used to obtain surface tension values by the drop-volume method[2], by comparison of drop volumes for distilled water and for 1% solutions of the surface-active agents. Interfacial tensions were measured in a similar manner, except that the hypodermic needle was bent into

a U shape, and the formation of drops of white spirit observed under the surface of an aqueous solution of the surface-active agent under test.

(c) Quantitative Estimation of Dye Fixation

Fixation was estimated by dissolution of the print in concentrated sulphuric acid by the method described for vat dyes by Michie and Thornton [3], followed by spectrophotometric measurement of the diluted extract.

(d) Decomposition of Sodium Formaldehyde-sulphoxylate on Storage of Prints

Sodium formaldehyde-sulphoxylate was estimated by the method described by Thornton and Liquorice [4], by treating measured areas of print with aqueous formaldehyde acidified with hydrochloric acid and titrating with standard iodine solution.

(e) Viscosity Measurements

The rheological behaviour of thickenings on dilution was determined with a Brookfield viscometer, using the No. 4 spindle at a range of spindle speeds. The Ferranti–Shirley (cone and plate) viscometer was used to obtain detailed viscosity–shearing stress relationships.

3. RESULTS AND DISCUSSION

(a) Properties of Emulsifying Agents for Oil-in-water Emulsions

For the emulsion to be stable, the disperse phase must be in the form of very small droplets, and the emulsifying agent must also be compatible with the electrolytes and dyes to be used with the emulsion.

Surface-tension Effects— Table I shows the effect of a number of surface-active agents on the surface tension of water and on the interfacial tension between white spirit and water.

TABLE I

Surface and Interfacial Tensions obtained with 1% Solutions of Surface-active Agents

Surface-active Agent	Surface Tension (dynes/cm.)	
	Air Interface	White Spirit Interface
Water	72·8	37·2
Lissolamine A (ICI)	47·9	3·54
Dispersol VL (ICI)	45·5	3·08
Turkey Red Oil PO (ICI)	45·3	5·55
Perminal PW (ICI)	35·0	1·60
Sodium lauryl sulphate	34·1	2·18
Lissapol N (ICI)	31·6	1·53
Aerosol 590 (Aero Chemicals Ltd., London)	29·3	0·66

The actual reduction in surface tension caused by the addition of a surface-active agent depends upon its concentration, but the effect is usually independent of concentration when more than 0·1–0·2% of the agent is present [5]. The measured values given in Table I are typical of those observed with the majority of surface-active agents.

From the table it can be seen that some surface-active agents can reduce the interfacial tension between the oil and water phases to a very low value, and the use of such agents in emulsions leads to good emulsion stability. In practice it has been found, for example, that emulsions formed with Turkey Red Oil PO are considerably less stable than those formed with, say, Lissapol N as emulsifier, which is in agreement with the interfacial tension values.

Effects due to Size of Droplets— The degree of mechanical agitation necessary to produce the very small droplets of disperse phase required for a stable emulsion depends on the nature of the emulsifying agent, but does not correspond to its relative effect on the interfacial tension. The viscosity of the emulsion also depends upon the oil droplet size and its distribution [6]: the smaller the disperse-phase droplets for a given phase volume ratio and the more uniform the size distribution, the greater the viscosity. The stability of emulsions formed using various classes of emulsifying agents has been studied by King and Mukherjee [7], who found with ionic agents that the presence of oil droplets greater than 7·5 μ. in diameter led to coalescence of the drops with loss in stability.

(b) The Use of Film-forming Agents in the Emulsion System

The film-forming agents added to the emulsion are usually of a similar nature to the thickenings used in textile printing, but the concentrations in the emulsion thickening are much less than would normally be used in a conventional gum thickening. The increase in viscosity that these agents give to the aqueous phase has a marked effect upon the viscosity of the resulting emulsion. This is shown in Fig. 1 for sodium alginate as the film-forming material.

Initial relatively small increases in the viscosity of the aqueous phase result in a large increase in the viscosity of the derived emulsion. Thus the use of small quantities of film-forming agents of this type, as long as the concentrations in which they are present do not detract from the technical advantages (mainly due to the low solids content) derived from the use of the emulsion thickening, may also give economic advantages in some cases.

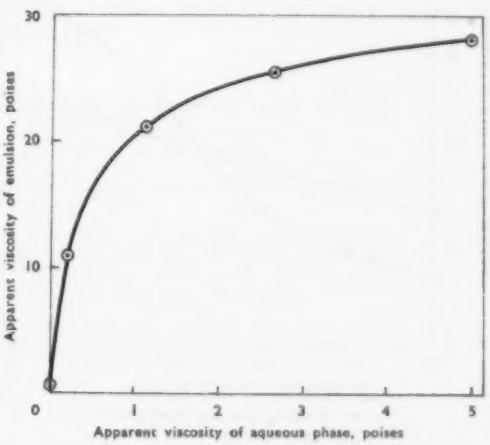

FIG. 1— Effect of External Phase on Emulsion Viscosity

Certain film-forming agents may also act as protective colloids for the emulsion, thus increasing its stability. In other cases the film-forming agent itself may be surface-active, e.g. cellulose ethers, although not sufficiently active for the emulsifying agent to be omitted. In the extreme it is possible to have a film-forming agent which also acts as the emulsifying agent (e.g. ammonium caseinate).

(c) The Rheology of Emulsion Thickenings

Emulsion thickenings derive their viscous nature from the resistance to flow encountered between the spherical droplets of the disperse phase, due partly to the electrical forces acting between them, and this is dependent, to some extent, on the emulsifying agent used. This is different from natural gums, for example, in which molecular chains are involved and at least part of the force acting between these molecular chains is hydrogen bonding. Natural gums show decreasing viscosity with increasing rate of shear owing to the progressive alignment of the molecular chains along the direction of flow. Emulsion thickenings, on the other hand, show a similar effect owing to progressive distortion of the spherical droplets of the internal phase as the rate of shear is increased. It is perhaps not surprising, therefore, that the variation of viscosity with dilution and rate of shear observed with emulsion thickenings has been found to differ from that observed with natural thickenings.

Variation of Viscosity with Concentration— Fig. 2 shows the variation in viscosity of a typical emulsion thickening, obtained using an ionic dispersing agent (Perminal PW), with concentration of white spirit in the emulsion, compared with that of a natural gum (tragacanth). It will be seen that, whereas the logarithm of the viscosity of the natural thickening decreases linearly with dilution, a sigmoidal relationship exists between these values for the emulsion thickening. Similar curves were obtained with emulsions formed with other ionic emulsifying agents. The use of non-ionic emulsifying agents (Dispersol VL and Lissapol N), on the other hand, gave curves similar to those for the natural thickenings, except that the rate of reduction in viscosity with increasing dilution was greater with the emulsion thickening (Fig. 3). These results show that, in practice, care should be taken in assessing, from a purely visual examination of a stock emulsion thickening, the dilution required to produce a print paste of satisfactory viscosity.

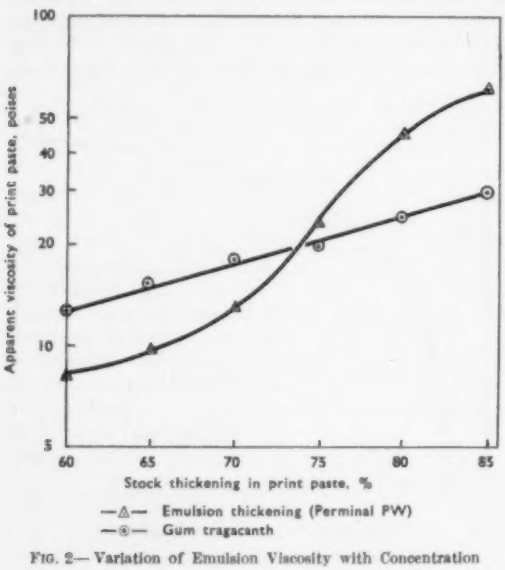

FIG. 2— Variation of Emulsion Viscosity with Concentration

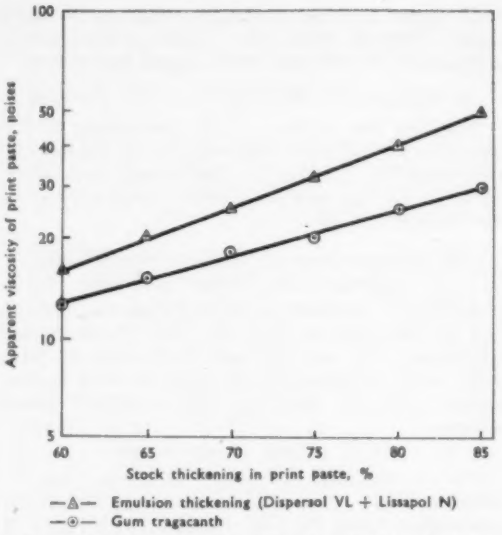

FIG. 3— Variation of Emulsion Viscosity with Concentration

It has been shown [8] theoretically that the variation of the viscosity of the emulsion η' relative to that of the external phase η_e is given by the equation—

$$\log \frac{\eta'}{\eta_e} = k\sigma$$

where k is a constant and σ is the phase volume relationship. This is only one of many equations proposed to define the viscosity of an emulsion system. Since $\log \eta$ decreases linearly with dilution, a plot of $\log \eta'$ against σ (or degree of dilution) should be linear. Fig. 2, therefore, shows to what extent an emulsion thickening may deviate from this relationship.

Variation of Viscosity with Rate of Shear— A liquid whose viscosity is independent of the rate of shear is said to exhibit Newtonian flow. Some liquids show a decrease in viscosity with increasing rate of shear, and such liquids are said to be *pseudo-plastic*. Others show increase in viscosity with increasing rate of shear, and these are said to be *dilatant systems*. Both pseudo-plastic and dilatant systems may be said to exhibit anomalous flow properties.

The flow properties of a range of natural thickenings have been examined [9] and related to the furnishing properties of the print paste and the definition and levelness of the resulting prints. It has been concluded that the definition of the prints depends on the paste maintaining a sufficiently

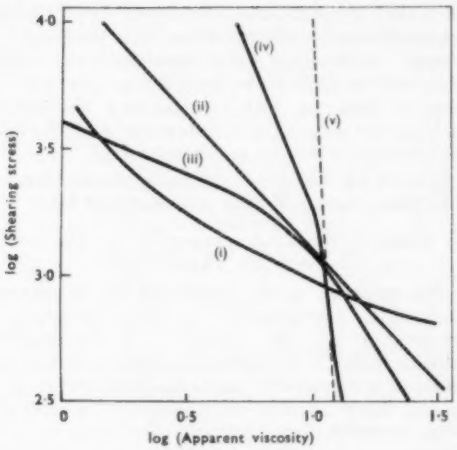

FIG. 4—Rheological Behaviour of Various Thickenings

high viscosity under the very high shear stresses operating at the nip of the printing roller. Shearing stress is given by the expression—

Rate of shear (sec.$^{-1}$) × Apparent viscosity (poises) = Shearing stress (dynes/cm.^{2})

By indirect means it has been estimated [9] that the shearing stress operative at the nip of the printing roller is about 4,500 dynes/cm.^{2}. Fig. 4 shows the variation of viscosity with rate of shear, measured with the Ferranti–Shirley viscometer, for some conventional thickenings and both a water-in-oil and an oil-in-water emulsion thickening.

From the curves it can be seen that—

(i) A low-viscosity alginate (Manutex SX/LD) behaves in an anomalous manner but exhibits near-Newtonian flow properties. (British gum gives similar curves.)

(ii) Oil-in-water emulsions show much less deviation from Newtonian flow properties than do water-in-oil emulsions.

(iii) At a shearing stress of about 2,000 dynes/cm.^{2} the alginate thickenings show a marked change in degree of anomaly similar to that observed with pigment dispersions [10]. This change is more marked with the high-viscosity type (Manutex RS) than with the low-viscosity type (Manutex SX/LD). Oil-in-water emulsion thickenings show no such break in their viscous behaviour.

The practical implication of these results is that an oil-in-water emulsion thickening which appears on hand stirring to have a similar viscosity to a print paste thickened with Manutex RS may when machine-printed be so viscous relative to the alginate thickening that fine lines on the pattern do not "print out". This effect shows itself in fine line designs as a discontinuity of the lines corresponding to the engraving marks on the printing roller. This has been overcome partly by increasing the nip pressure on the printing roller but more especially by using less viscous print pastes, which thus appear to be relatively thin on hand stirring, although the paste prints satisfactorily. A reduction in viscosity also yields more level prints. This phenomenon is well known to printers using conventional thickenings. For example, it is known that a "thin" (i.e. dilute) British gum paste gives a much sharper mark than a thin paste of a starch–tragacanth thickening (which shows greater anomaly than British gum).

One point which is of interest is that the emulsion shows approximately the same degree of anomaly whether or not a high- or a low-viscosity alginate is used as the film-former, even if the proportion of the alginate in the emulsion is increased threefold.

4. PRACTICAL APPLICATION OF EMULSION THICKENINGS IN TEXTILE PRINTING

Examination of a wide range of emulsifying agents and film-forming materials has enabled suitable recipes to be selected for the application of a number of different classes of dyes (see Table II).

Emulsion thickenings may be used for both screen and roller printing, but to date no satisfactory emulsion has been obtained for conventional block printing. Where a polyurethane foam replaces a conventional block surface, however, transfer of an emulsion thickening may be obtained [11].

TABLE II

Survey of Oil-in-water Emulsion Thickenings suitable for Various Classes of Dyes

Class of Dye	Film-forming Agent	Emulsifying Agent	Additional Properties of the Emulsion
Reactive	Manutex RS Celacol MM 10	Perminal PW	Also of interest for applying disperse dyes
Vat	Manutex C Celacol M 2500 Solvitose C 5	Perminal PW Perminal EML	May be used for white discharges (omitting the alkali carbonate). An addition of Bedafin 285X (ICI) may be required
Onium	Indalca U Meypro Gum CRX Gum tragacanth	Dispersol VL+ Lissapol N	Also of interest for acid dyes, stabilised oxidation blacks, and azoic diazo components
Azoic	Manutex C Celacol M 2500 Meypro Gum CRX Gum tragacanth	Dispersol VL+ Lissapol N	Of interest for neutral-steaming stabilised azoic combinations

(*a*) *General Advantages arising from the Use of Emulsion Thickenings*

(i) Printing is possible from a thickening of very low solids content even under conditions where conventional thickenings of high solids content would normally be necessary. This property, which is characteristic of emulsion thickenings, is due to the contribution made to the viscosity of the print paste by the volatile hydrocarbon. Some 35–65% of an emulsion print paste consists of the hydrocarbon oil, while the gum solids content may be as low as 0·3%. Conventional gum-thickened print pastes may, on the other hand, contain as much as 30% gum solids.

(ii) As a consequence of their low solids content and the volatile nature of the oil phase, emulsion thickenings dry rapidly when printed on the fabric. This is of particular importance in screen printing.

(iii) Fabrics printed with emulsion thickenings are very much softer after drying than are those to which a conventional print paste has been applied. In this condition they are much less liable to creasing and/or marking off.

(iv) The residual solids remaining on the printed fabric after the dye has been fixed are more readily removed by washing, since the film-forming material is present in very much lower concentrations than when normal thickenings are used. This allows more rapid and less severe washing-off treatments to be given to the fabric while ensuring an excellent handle in the finished print with minimum distortion of the fabric surface. This is particularly valuable in the case of raised or pile fabrics, e.g. light-weight dress corduroys. Where wet processing includes a piling operation, the danger of marking-off from the printed design is much reduced when emulsion thickenings are used.

(v) The absence of a residual thickening film after washing and drying often results in brighter prints, and in some cases quite a striking improvement results. The soft handle of washed prints ensures a very even pick-up of resin and avoids the necessity of giving a malting or other enzymic treatment to printed goods prior to resin finishing.

(vi) The emulsion thickening generally penetrates less deeply into the fabric than many conventional thickenings, thus giving a higher visual colour yield. Although quantitative measurements show that in some cases the overall dye transfer is not significantly different from that occurring with normal thickenings, the emulsions do enable economies in dyes to be made, since the apparent yield is high, as with starch-based thickenings, without the associated disadvantages of the latter with regard to handle and washing off.

Specific advantages of emulsion thickenings with individual classes of dyes are described later.

(*b*) *General Printing Properties of Oil-in-water Emulsion Thickenings*

The printed "mark" produced by an emulsion thickening, particularly in roller printing, is determined by the viscosity of the made-up printing paste. In machine printing, it has been found that more level prints are obtained if rollers having fairly shallow engravings (0·15–0·20 mm., 0·004–0·006 in.) are used and the prints are given a light crush. This is particularly important where a blotch pattern is being printed.

The viscosity of an emulsion print paste is mainly controlled by the quantity of dispersed "oil" (i.e. white spirit or distillate) present, but the film-forming material added also contributes towards the final print paste viscosity. Thus, if a print paste proves too thin for satisfactory printing, the viscosity may be raised either by adding further quantities of fresh stock thickening and adjusting the dye strength accordingly, or by increasing the amount of film-forming material in the recipe.

Should frothing of the emulsion prove troublesome at any stage, this can be overcome by the addition of an anti-foaming agent. Some anti-foaming agents tend to break emulsion thickenings, but Silicone Anti-foam M 435 (ICI) is very satisfactory in this application.

Emulsion thickenings prepared by the recipes described below are very stable under normal colour shop conditions. On prolonged storage, however, some separation of the oil phase may occur. If this occurs, stirring or rehomogenising the emulsion will produce a thickening of unimpaired performance. Heating tends to break down emulsion thickenings, and they should normally be stored in a cool place, preferably in non-absorbent containers fitted with lids. Storage in uncovered containers causes some emulsion

TABLE III

Typical Recipes for the Preparation of Stock Emulsion Thickenings

Dye class	Reactive		Vat	Onium	Azoic
Thickening	A	B	C	D	E
Manutex RS (5% soln.)	10	10	—	—	—
Manutex C (5% soln.)	—	—	10	—	10
Indalca U (8% soln.)	—	—	—	7½	—
Perminal PW (10% soln.)	10	—	15	—	—
Lissapol N	—	5	—	5	5
Dispersol VL	—	7½	—	7½	7½
White spirit (distillate, etc.)	80	77½	35	80	77½
Potassium carbonate	—	—	12	—	—
Sodium formaldehyde-sulphoxylate	—	—	10	—	—
Water	—	—	18	—	—
	100	100	100	100	100
Rate of usage (lb./100 lb. print paste)	60–70		80	65–75	60–70

TABLE IV

Summary of Printing Applications of Emulsion Thickenings

Dye Class	Dyes suitable for Application with Emulsion Thickening	Applications for which Emulsions are of Special Interest	Specific Advantages of Emulsions over Conventional Thickenings
Reactive	All Procion dyes except Procion Black HG suitable with Thickening A (Table III). Thickening B may be used with all Procion dyes Print pastes are prepared as recommended [13]	For fixation alongside pigments by dry heat treatment (i.e. baking) [13] For printing viscose rayon tufted carpets [14], where ease of washing-off is a considerable advantage	Increases in visual colour yield of up to 20% obtained, particularly on viscose rayon fabrics and with designs receiving a crush on the printing machine. Procion Brilliant Blue H7G gives brighter and much stronger prints, showing marked improvement on viscose rayon fixed under dry steaming conditions
Vat	All Caledon and Durindone vat dyes suitable except— Caledon Printing Black R Paste QF Caledon Printing Black 2R Paste Caledon Printing Yellow 6G Paste (C.I. Vat Yellow 27) Caledon Printing Blue XRN Paste (C.I. Vat Blue 4) Where available, QF-quality dyes are preferable	For printing raised or pile fabrics, where soft handle of the finished goods is essential. Also useful when printed fabric is to be resin-finished. Excellent results in screen-printed designs with high coverages	Gives high visual colour yields, like starch-based thickenings, without the disadvantages of the latter In certain cases, prints appear notably brighter, e.g. with— Caledon Printing Jade Green XBN (C.I. Vat Green 1) Durindone Printing Pink FF (C.I. Vat Red 1)
Onium	All Alcian X dyes suitable using any of the recommended recipes [15]	For non-steam styles with Alcian Blue 8GX (C.I. Ingrain Blue 1) alongside pigments, using the acetic acid–sodium acetate recipe	Prints generally appear brighter and have a superior handle to those obtained with starch-based thickenings
Azoic	The following Brentogen dyes are suitable when applied by the acid-age process— Yellow G (C.I. Azoic Yellow 1) Red R (C.I. Azoic Red 2) Bordeaux R Red Violet 2R Violet B Blue B Navy Blue B (C.I. Azoic Blue 9)	For styles where Brentogens may be used to supplement pigments to give certain colours of superior dry-cleaning fastness. For certain markets, the washing-off process may be omitted if an emulsion thickening is used	Where mixed Brentogen–pigment styles are being operated, fewer compatibility difficulties arise

thickenings to dry out on the surface, but if such conditions are encountered, drying out may be minimised by adding about 5% urea to the stock thickening.

(c) *Application of Emulsion Thickenings with Individual Classes of Dye*

Table III gives some typical recipes for the preparation of stock emulsion thickenings. They are given on a weight/weight basis, since the specific gravity of the emulsion depends on the type of hydrocarbon oil used and on the degree of emulsification. Preparation of an emulsion thickening on the more conventional weight/volume basis could lead to variation in the effective percentages of the components of the emulsion.

The general method of preparing the stock emulsion thickening is to mix the film-forming material and emulsifying agent and to add the hydrocarbon oil, in portions, to this mixture with constant stirring. Time should be allowed for each fresh addition of hydrocarbon oil to emulsify before further additions are made. The emulsion is finally passed through a homogeniser (e.g. of the Rapisonic type).

The dyes which are suitable for application with the above emulsion thickenings are summarised in Table IV, together with those applications which are of special interest using the emulsion technique. Some indications of the specific advantages of using an emulsion in place of a conventional thickening are also given.

The recipe given in Table IV for the stock emulsion thickening for printing vat dyes does not contain glycerol, as such additions have much less effect than with normal thickenings applied to cotton fabrics, even with very dry steaming conditions. There appears to be an advantage in yield on viscose rayon if an addition of 5% glycerol on the weight of print paste is made. Although the visual colour yield of many vat dyes applied from emulsions is not in general increased, addition of glycerol does increase the fastness to rubbing of the prints, particularly if the dyes are applied by screen printing. The rate of reduction of vat dyes is lower when printed from emulsion thickenings than when printed from natural gum thickenings, and this is illustrated in Fig. 5. For this reason, it is preferable to use QF quality dyes whenever possible. No particular advantage has been found in applying vat dyes from water-in-oil emulsions by the pad–flash age development process [16], although such a process has been exploited by du Pont in the U.S.A. (Savagraph process).

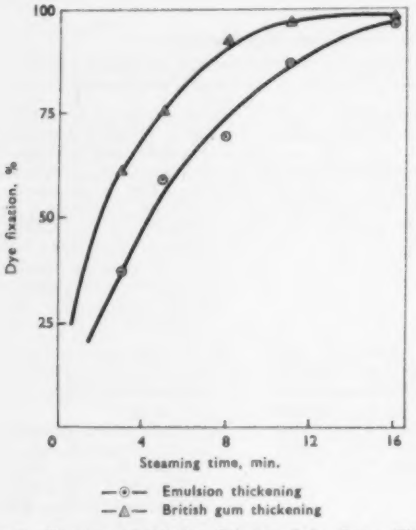

FIG. 5—Fixation of Caledon Printing Jade Green XBN (C.I. Vat Green 1)

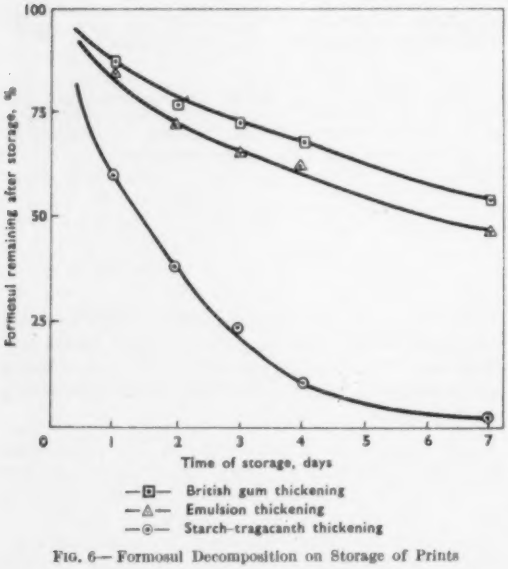

FIG. 6—Formosul Decomposition on Storage of Prints

In screen printing under warm, humid conditions, emulsion prints with vat dyes show much less tendency for the sodium formaldehyde-sulphoxylate to decompose than when a starch–tragacanth thickening is used. This is rather surprising in view of the low solids content of the thickening. This behaviour is also an advantage if the steaming of roller prints has to be delayed for some reason or other. The rates of decomposition of sodium formaldehyde-sulphoxylate in various thickenings are shown in Fig. 6.

Discharge Styles—Semi-bulk-scale trials have shown that it is possible to use an emulsion-thickened print paste of the type recommended for vat dyes (Table III, Thickening C, omitting the alkali carbonate and diluting with water) in place of a conventional high-solids thickening to obtain white discharges. Under the steaming conditions used in these trials, good definition was obtained using this emulsion, but printers may find it beneficial to add a small quantity of Bedafin 285X to the print paste to maintain the printed mark if wet steaming conditions are encountered. The Bedafin 285X is added at 1% on the weight of print paste by diluting the resin with an equal volume of toluene and emulsifying this solution directly into the thickening. Successful working of this process will depend on local steaming conditions and on the type of fabric being printed. Additions of Bedafin 285X lower the stability of the emulsion discharge paste, and initial trials should be carried out in the absence of this additive.

Coloured discharges using vat dyes may also be obtained using the emulsion thickening recommended for these dyes. In this case, Bedafin 285X should not be added, as this tends to break the vat emulsion print paste. Semi-bulk-scale trials have shown that less haloing is present with an emulsion discharge paste than when a British-gum vat thickening is used under comparable conditions.

Printing of Other Dye Classes and Non-cellulosic Fibres with Emulsion Thickenings—For printing fibres such as nylon, secondary cellulose acetate, cellulose triacetate, acrylic, and polyester fibres, emulsion thickenings give good clarity of mark, normally associated with high-solids thickenings, while at the same time they do not exhibit the poor adhesion properties of the latter. To obtain optimum clarity, however, it is advantageous to increase the concentration of film-forming agent in the emulsion recipes above that required for the printing of cellulosic fibres. Experience in the laboratory has shown that, during handling after printing and drying, there is less tendency for the thickening from the emulsion to flake off, resulting in specks of colour developing on the white portions of the fabric on steaming. For example, disperse dyes may be applied to nylon, cellulose acetate, or triacetate fabric using the emulsion recipe recommended for Procion dyes (Table III, Thickening A), while acid and mordant dyes may be applied using the emulsion recommended for Alcian X dyes (Thickening D). The latter emulsion recipe is satisfactory also for printing oxidation blacks such as Soloxan Black SN 150 (C.I. Oxidation Base 3), and for printing Fast Colour Salts (ICI) on prepared cloth. Neutral-steaming stabilised azoic combinations may also be printed on cellulosic fibres using the emulsion recipe recommended for Brentogens (Thickening E), but with those dyes the use of such solvents as Cellosolve or alcohol should be avoided, as they cause the emulsion to break.

On union fabrics such as Terylene and cotton (66–34 type) and Courtelle and cotton (50–50 type), vat dyes show marked increases in visual yield, compared with conventional thickenings, when applied from an emulsion thickening.

5. CONCLUSION

The foregoing recipes and application details should be regarded only as general indications of the fields in which emulsion thickenings may be used to advantage. If care in their selection is exercised, other emulsifying agents and film-forming materials may be used, as may be determined by trials carried out under local conditions. Nevertheless, adherence to the recipes given is suggested, at least in preliminary trials, particularly for those unfamiliar with emulsion technology.

While it is not suggested that emulsion thickenings should replace all conventional thickenings used in textile printing, there would be considerable advantage in their use to solve specific problems associated with the latter type. Finally, it might be borne in mind that, apart from the technical advantages which accrue from the use of emulsion thickenings, comparative costings show that, in general, emulsion thickenings are no more expensive, and in some cases are considerably cheaper, than conventional thickenings.

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(*MS. received 20th March 1959*)

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Fast Dyeings on Wool–Cellulosic Unions

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Meetings of the Scottish Section held at the St. Enoch Hotel, Glasgow, on 9th December 1958, Mr. W. G. B. Grant in the chair; of the West Riding Section held at the Victoria Hotel, Bradford, on 26th February 1959, Mr. R. Heaton in the chair; and of the Huddersfield Section held at Silvio's Café, Huddersfield, on 17th March 1959, Mr. H. Boothroyd in the chair

In recent years, increased interest in wool–cellulose unions has, in some cases, called upon the dyer to produce the same standards of fastness as can be obtained on the separate fibres. It has been found that by careful selection of dyes and dyeing methods, it is possible to apply reactive, onium, vat, and azoic dyes satisfactorily to wool–cellulose unions without causing significant fibre damage.

In the post-war years the number of man-made textile fibres has increased considerably, and there has been an increasing tendency for manufacturers to use these in blends with one another and in blends with wool or cotton. The price of wool in recent years has been high, and large quantities of blends of wool with cotton or viscose rayon have been manufactured, primarily to obtain a cheaper product than can be obtained from wool itself. In addition to this, for many years there have been a number of materials traditionally made from wool and cellulose, such as blazer cloths consisting of a cellulose warp and a woollen weft, as well as light-weight blended wool–cellulose unions for the shirting and pyjama trade.

The dyer is, therefore, often called upon to produce the same colours and fastness properties on wool–cellulose unions as can be obtained on the separate fibres. Clearly, the simplest method of obtaining the desired colour and fastness properties is to dye the individual fibres before blending, for the following reasons—

(1) Dyeing of the wool and the cellulose may be carried out under the optimum conditions of pH, temperature, etc.

(2) Dyes may be easily selected to give the requisite fastness properties

(3) Extremely high standards of levelness and colour matching are not required, since correction is easily effected during normal blending and carding.

Whilst from the dyeing point of view this is the most efficient method of colouring wool and cellulose for the production of unions, there are obvious economic and commercial advantages accruing from dyeing the union as yarn or cloth. The spinning and other preparative processes are simplified, and the material may be coloured as desired at a later stage.

In the dyeing of any wool–cellulose union, the result depends on the relative behaviour of the fibres towards the dyes used, and the production of solid colours depends either on (*a*) the selective dyeing of each fibre with its own class of dyes or (*b*) the choice of dyes which will dye both fibres.

The most commonly used method is to employ either direct dyes possessing union-dyeing properties or a suitable mixture of acid and direct dyes. This method, whilst relatively simple, cannot produce dyeings possessing the highest wet fastness properties and also suffers from a number of defects. It is impossible to obtain the same brightness on wool–cellulosic unions as can be achieved with the brightest acid dyes on wool,

since direct dyes as a class are duller than acid dyes. This position is further complicated by the fact that there is always some staining of wool by direct dyes, this staining usually being duller than the colour on cellulose.

Whilst it is generally possible to obtain dyeings of satisfactory light fastness on wool unions with normal union dyes, the production of dyeings of high wet fastness is limited by the wet fastness obtainable on the cellulose with direct dyes. Although the introduction of various products and methods for aftertreating direct dyes to improve their wet fastness has undoubtedly been a great advance, there are still many cases where the use of wool–cellulose blends is severely restricted by the limited wet-fastness properties which can be obtained.

A study of both technical and trade literature has revealed an absence of any really significant work on the production of dyeings on wool–cellulose union materials possessing the highest wet-fastness properties. The purpose of this lecture is to describe recent work done on this problem in the laboratories at Blackley.

A consideration of the ranges of dyes available for dyeing cellulose to higher wet fastness than that obtainable with direct dyes, either straight or aftertreated, shows the following general types—sulphur, reactive, onium, vat, azoic, and solubilised vat dyes—and it has been found that, by careful selection of dyes and dyeing methods, the above classes, with the exception of sulphur and solubilised vat dyes, may be applied under suitable conditions to wool–cellulosic unions. The sulphur and solubilised vat dyes have been found unsuitable for a number of reasons, among the more important of which are severe or incipient damage to the material and, with the latter class, the extreme difficulty of achieving complete oxidation of the dye on the wool portion of the union. For these reasons, therefore, these two classes must be considered of no practical interest for dyeing these types of material.

In the application of the other four dye classes, it cannot be stressed too strongly that the production of solid dyeings is almost entirely dependent on the type of material to be dyed: variations in the amounts of wool and cellulose, cloth and yarn constructions, treatments before dyeing, and the depth required—all exert some influence on solidity. For example, some dyes give satisfactorily solid dyeings in pale depths, but in heavy depths may dye one fibre much more heavily than the other; and a combination which gives a solid on wool–cotton may be completely unsatisfactory on a similarly constructed wool–viscose rayon union. It is, therefore, desirable to prepare small-scale laboratory dyeings prior to carrying out bulk dyeings when new colours or different qualities of materials are encountered.

Effect of Dyebath Conditions on Physical Properties of the Material

One of the first considerations facing the dyer is the condition of the material after dyeing. Since wool is normally considered to be extremely sensitive to the action of alkalis, and the application of reactive, vat, and azoic dyes requires the use of alkaline agents, tests have been carried out to determine the effect of various treatments on the strength of wool–cotton union yarns.

Cheeses of a 50 : 50 wool–cotton union yarn were treated in blank dyebaths containing varying amounts of caustic soda alone and in the presence of sodium hydrosulphite to simulate the conditions likely to obtain in azoic and vat dyeing. In addition, the effect of adding a diazotised base was studied. The use of acetic acid followed by soda ash as in Alcian dyeing, and the use of common salt and trisodium phosphate for Procion dyes, were examined. As controls, a sample of untreated yarn and a sample treated with 30% Glauber's salt (on the weight of material) at 95°c. for 1 hr., as in conventional union dyeing, were included.

After treating under the different conditions, the material was given a rinse in cold water for 5 min. and then dried, after which the breaking load was measured. The yarn was conditioned at 65% R.H. and 21°c. for 24 hr. before testing, and the breaking load measured by means of a single-thread pendulum tester of capacity 500 g., operating under constant-rate-of-traverse conditions (12 in./min.). The results obtained are given in Table I.

It will be seen from the figures given in this table that treatment 12 (Soledon dyeing method) results in an appreciable decrease in both count × strength product and extensibility of the yarn. Thus, apart from any question of difficulties in application, this method must be considered unsatisfactory from its effect on the physical properties of the material. In none of the other cases is there any significant effect on these yarn properties, and hence the treatments should be capable of application under practical conditions without having any seriously disadvantageous effect on the material.

These tests have been carried out on a blended wool–cotton union yarn rather than on separate wool and cotton yarns, since under practical conditions it is the effect on the whole blended material which is important. If there is some degradation of the wool, the latter will probably be supported by the cellulose. In an attempt to throw some light on this possibility, abrasion tests, using a Martindale wear tester, were carried out on a sample of untreated 50:50 blended wool–cotton union cloth and on similar material dyed with three azoic combinations and three vat dyes.

It will be clear from these results (Table II) that the abrasion resistance of azoic-dyed material is not significantly reduced from that of the undyed material. Taken in conjunction with the strength figures of Table I, they show that azoic dyeing does not affect the physical properties of wool–cotton union materials. On the other hand, vat-dyed material shows a significant loss in abrasion resistance, although no loss in tensile strength occurs. This seems to suggest that there may be a decrease in wear resistance of vat-dyed union materials, although the normal strength is unaffected. This probably arises from incipient damage to the wool portion of the union, and the subsequent easier removal of the wool during abrasion.

TABLE I

Effect of Dyebath Conditions on Strength of 50:50 Wool–Cotton Union Yarn

No.	Treatment	Concn. (g./litre)	Temp. (°c.)	Time (min.)	Extension (%)	Count (woollen)	Breaking Load (oz.)	Count × Strength Product
1	Untreated yarn (control)				7·2	50·9	11·3	575
2	Glauber's salt (normal union-dyed control)	30%	95	60	7·1	48·7	11·0	536
3	Caustic soda*	4	40	45	7·4	48·6	11·6	564
4	Caustic soda	4						
	+ Hydros	4	40	45	7·3	46·1	11·4	526
5	Caustic soda	6						
	+ Hydros	4	40	45	7·1	52·3	10·6	554
6	Caustic soda	2						
	+ Hydros	4	40	45	7·3	51·3	10·6	544
7	Caustic soda	4	Cold	60	7·6	49·9	11·5	574
8	Caustic soda	4	Cold	30				
	then diazotised Brentamine Fast Red B Base (C.I. Azoic Diazo Component 5)	3	Cold	30	7·0	48·3	10·9	526
9	Caustic soda	4	Cold	30				
	then diazotised Brentamine Fast Red KB Base (C.I. Azoic Diazo Component 32)	3	Cold	30	6·6	53·4	10·6	566
10	Acetic acid (30%)	10	Cold	20				
	then raise to		100	30				
	and soda ash	3						
	+ Lissapol NC	3	60	30	7·6	47·7	12·0	572
11	Salt	40						
	+ trisodium phosphate	6	Cold	75				
	rinse and							
	Lissapol NC	3	95	15	6·0	53·3	11·5	613
12	Glauber's salt	40	50	30				
	then H_2SO_4	10						
	+ NH_4CNS	2%						
	+ $Na_2Cr_2O_7$	3%	50	20	5·5	50·6	9·6	486

* Caustic soda flake was used in all cases.

TABLE II

Abrasion Tests on 50:50 Blended Wool–Cotton Cloth

Treatment	No. of Rev. to hole Fabric
Undyed material	7,000
Brenthol BC + Brentamine Fast Red AL Salt (C.I. Azoic Coupling Component 26 + C.I. Azoic Diazo Component 36)	6,000
Brenthol BC + Brentamine Fast Red TR Salt (C.I. Azoic Coupling Component 26 + C.I. Azoic Diazo Component 11)	6,500
Brenthol CT + Brentamine Fast Red TR Salt (C.I. Azoic Coupling Component 8 + C.I. Azoic Diazo Component 11)	7,500
6% FD Caledon Gold Orange 3G (C.I. Vat Orange 15)	4,500
5% FD Caledon Pink RL (C.I. Vat Red 43)	3,500
2% FD Caledon Jade Green XN (C.I. Vat Green 1)	4,000

Attention was, therefore, turned to the practical problems associated with the application of the faster types of dyes for cellulose to wool–cellulose unions, and these will be dealt with separately in the following order— Procion dyes, Alcian dyes, vat dyes, and azoic dyes.

Procion Dyes

The Procion dyes are recommended for the production of bright colours of pale–medium depth with high light and wet fastness on cellulosic fibres. They therefore offer a means of achieving high wet-fastness properties on the cellulose portion of wool–cellulose unions by a simple dyeing method. When applied by the normal cold-dyeing technique, the Procions dye the cellulose in the union, leaving the wool only slightly stained. Raising the temperature results in some increase in adsorption of Procion by the wool, but under normal conditions solidity is never achieved, the cellulose always being dyed more heavily than the wool.

Under any given set of dyeing conditions the staining of the wool depends upon the extent to which the wool fibre has been degraded, increased damage resulting in heavier staining. Thus, in the case of unions of cellulose and reclaimed wool, the amount of Procion adsorbed by the protein fibre will be considerably greater than in the case

of a union containing virgin wool. Similar effects are obtained when chlorinated wool is incorporated in the union, the staining depending on the extent of chlorination and also the exact method of treatment. It is possible, provided that the damage is great enough, to arrive at a condition where the protein portion of a union is dyed more deeply than the cellulose. If such a state is reached, the use of Procion dyes is to be avoided, since there is then no means of "filling in" the cellulose to the requisite standard of wet fastness.

Under normal conditions, where the cellulose will be dyed more deeply than the wool, the latter may then be filled in with Carbolan or Coomassie dyes. This may be carried out in the Lissapol NC soaping bath used for the Procions or in a fresh bath using Glauber's salt or ammonium sulphate as assistant. Experience has indicated that this technique may be applied to a wide variety of wool–cellulose union yarns and fabrics. In the case of yarns, package or hank dyeing may be employed, and in the case of piece goods, winch dyeing; pad–jig or pad–winch develop and in certain cases a pad–batch method may be employed. The following cold-dyeing Procion dyes may be used for dyeing the cotton or viscose rayon portion of union materials—

Procion Yellow RS
Procion Brilliant Yellow 6GS
Procion Brilliant Orange GS
Procion Brilliant Red 2BS
Procion Brilliant Red 5BS
Procion Scarlet GS
Procion Blue 3GS
Procion Brilliant Blue RS

The following general methods have been developed and may be taken as a guide for the application of Procion dyes to wool–cellulose unions—

1. PAD–JIG OR PAD–WINCH DEVELOP

The prepared material is padded through a solution containing—

Already dissolved dye	1–100*
Perminal PP	2·5
Common salt	10

at 85–100°F. The colour is then developed on the jig or the winch, according to the nature of the fabric, in a cold bath set with—

Soda ash	5–10*
Common salt	30–60

for 60 min. The goods are then rinsed and "soaped" in a bath set with Lissapol NC (3 parts*) at 190–205°F. for 15 min.

2. YARN AND PIECE DYEING FROM LONG LIQUORS (E.G. WINCH AND HUSSONG-TYPE MACHINES)

The prepared and thoroughly wetted-out material is entered into a cold dyebath set with the predissolved dye, and a total of 30–60 parts of common salt per 1,000 parts of liquor is added, in portions, over a period of 30 min. After a further 10 min. 1–5 parts of soda ash per 1,000 parts of liquor in the case of wool–viscose rayon (or 1–5 parts of trisodium phosphate in the case of wool–cotton) is added to the dyebath, and dyeing continued for a further 60 min.

The goods are then rinsed and soaped as above.

3. PAD–BATCH METHODS

The prepared material is padded cold through a solution of dye, alkali, and wetting agent, after which the padded material is batched for 1–24 hr. depending on the depth of colour and the alkali used, washed off, and soaped in the normal manner. The equipment required is simple, consisting of a padding mangle, a device for mixing dyes and alkali solution, a stillage for batching, and finally an apparatus for washing.

In general, the batching time depends on the amount and type of alkali used, the stronger the alkali the lower the batching time. For wool–cellulose unions both sodium bicarbonate and soda ash may be used as convenient alkaline media. The advantage of using bicarbonate lies in increased stability of the dye solution and improved penetration resulting from the slower rate of dye–fibre reaction.

4. "FILLING IN" THE WOOL IN THE SOAPING BATH

In cases where the wool is left weaker than the cellulose when dyed with Procion dyes and filling-in is to be carried out in the soaping bath, the material is entered into the bath set with 3 parts Lissapol NC per 1,000 parts of liquor at 100–110°F., and after running for 5 min. the pH is adjusted to 6–7 by addition of ammonium sulphate, monoammonium phosphate, or acetic acid. The requisite amount of predissolved milling acid dye is then added, and the temperature raised to 190–205°F. over 20–30 min. and held at this value for 30 min. The material is then rinsed and dried.

The following milling acid dyes have been found satisfactory for this application—

Carbolan Yellow 4GS	C.I. Acid Yellow 70
Carbolan Brown BLS	—
Coomassie Fast Scarlet BS	C.I. Acid Red 111
Coomassie Geranine 2GS	—
Carbolan Crimson 3BS	C.I. Acid Red 139
Coomassie Ultra Sky SES	C.I. Acid Blue 112
Coomassie Blue BLS	C.I. Acid Blue 59
Carbolan Brilliant Blue 2GS	—
Carbolan Brilliant Green 5GS	C.I. Acid Green 28
Coomassie Fast Grey 3GS	C.I. Acid Black 48

It is possible, by careful selection of milling acid dyes, to obtain not only solid colours on wool–cellulose unions but also tone-in-tone and contrast effects on piece goods or yarns where either the cloth construction or the blending lends itself to this application.

Alcian Dyes

The successful dyeing of cellulose with the Alcian X dyes to give bright blues and greens otherwise unobtainable with the same standard of fastness properties has prompted interest in their potential value for dyeing wool–cellulosic unions. Experience has shown that, with careful control, Alcian Blues 8GX (C.I. Ingrain Blue 1), 5GX, and

* All parts per 1,000 parts of liquor.

2GX may be applied successfully to the cellulose portion of wool–cellulose unions. In the case of Alcian Greens 2GX and 3BX (C.I. Ingrain Greens 2 and 1), any variation in colour reproduction which makes their application to cellulose of limited interest under normal conditions is of less importance, since it can be corrected when the wool is filled in.

As with other fast dyes, the distribution of Alcian dyes between wool and cellulose depends on the condition of the wool, ranging from a virtually undyed wool in the case of virgin fibres to a very heavy stain with badly degraded protein fibres. Another factor of considerable importance is the thorough preparation of the substrate before dyeing. Alcian dyes possess greater affinity for many of the impurities met with in grey goods than for cellulose itself; hence a thorough and uniform prepare is essential in the interests of both level dyeing and maximum fastness. It is also essential to remove all residual alkali, detergent, and anion-active agents prior to dyeing, as the presence of these types of product has an adverse effect on dyebath stability.

From tests on the dyeing of wool–cellulose piece goods, dyeing may be carried out on the winch in the case of woollen cloths, or preferably on the jig in the case of pale colours on light-weight materials. Yarn dyeing appears to be a practical proposition with both Hussong-type (preferably two-stick) and circulating machines. The following general dyeing method has been found to give satisfactory results under practical conditions—

The material is run in acetic acid and Lissolamine A 50% cold for 15 min. (see Table III).

TABLE III

Amounts of Acetic Acid and Lissolamine A 50% in Alcian Dyebaths

Depth (%)	Acetic Acid (80%) (g./litre)	Lissolamine A 50% (%)
0·25	4	2
0·75	3	1
1·5	2	–

The dye, previously dissolved in acetic acid, is added over 10 min., and dyeing allowed to proceed cold for a further 15 min. The temperature is raised to the boil in 30 min., and boiling continued for 30 min. After rinsing, the dye is developed by running with soda ash (3 g./litre) at 120–130°F. for 20 min. In heavy dyeings, in order to achieve the maximum fastness to rubbing, a "soaping" treatment with Lissapol NC (3 g./litre) at 120–130°F. for 15 min. is recommended. Filling-in of the wool portion of the union is then carried out in a fresh bath using the following selected dyes—

Coomassie Turquoise Blue 3GS	—
Lissamine Ultra Sky RS	C.I. Acid Blue 67
Coomassie Ultra Sky SES	C.I. Acid Blue 112
Coomassie Yellow 7GS	C.I. Acid Yellow 44
Coomassie Brilliant Blue G 250	C.I. Acid Blue 90

This is carried out by conventional wool-dyeing techniques, and the amounts of acid dye required will vary both with depth and with the quality of the union being dyed.

Vat Dyes

The next step in obtaining an improvement in wet or light fastness on cellulosic materials over the use of Procion or Alcian dyes is the use of vat or azoic dyes, which in addition increases the number of fast colours obtainable. Both classes derive their fastness properties from the production of insoluble pigment inside the fibre. Vat dyeings, obtained by the normal leuco method and soaped, are extremely resistant to removal by normal wet treatments, representing at present the highest standard of fastness achievable on cellulosic materials.

From tensile-strength measurements on union yarns it appears (Table I) that caustic soda and Hydros (both 4 g./litre) at 40°c. for 45 min. do not cause any marked degradation of the yarn. Although a caustic soda concentration of 6 g./litre under the same conditions of treatment also shows no significant loss in tensile strength, 4 g./litre has to be taken as the upper limit for practical usage.

It is well known that vat dyes are classified in their method of application by the amount of caustic soda required for the formation of a stable leuco dyebath and the temperature of dyeing, and there are very wide variations between individual members of the vat range in these respects. The limits of 4 g. caustic soda per litre and a dyeing temperature of 40°c. impose severe limitations on the choice of dyes, apart from any considerations of solidity of colour on wool and cotton or viscose rayon which may also arise. Many of the commonly used anthraquinone vat dyes for cotton or viscose rayon are unsuitable for application, since under these conditions it is not possible to obtain sufficiently stable leuco solutions or, in certain cases, to obtain the leuco compound at all. In general, selection of dyes must be made from those applicable to cellulose by method 2, but for certain colours (i.e. blues and greens) the use of selected method 1 dyes is unavoidable.

Experience in the laboratory and under bulk-dyeing conditions has shown that the dyeing properties of vat dyes, in common with those of other fast dyes, are affected by variations in the proportions of wool and cellulose, the cloth constructions, and the depth of colour required. Some dyes give satisfactory results in pale colours, but in heavy depths may dye the wool deeper than the cotton, or even give different hues on the two fibres. It is desirable, therefore, to prepare sample dyeings before carrying out bulk work when new colours or materials of different quality are encountered. Small differences in colour between the wool and the cellulose may generally be corrected in the soaping bath with selected direct or milling acid dyes.

The application of vat dyes is somewhat restricted and is generally confined to shirting or children's wear, where the highest washing fastness is essential. Again, in view of the fact that the dye solution is reduced and is unstable, being readily oxidised to the insoluble pigment, application is generally limited to wool–cellulose union yarns dyed in package form in circulating machines

or to piece goods dyed on the jig. In the latter case, therefore, the material must be capable of withstanding the mechanical stresses encountered in processing under these conditions. After dyeing on the jig, it is recommended that soaping or any topping required is carried out on the winch, as apart from more efficient removal of loose pigment, this treatment gives the cloth the opportunity to relax and releases tensions set up during the jigging operation.

From tests carried out over a range of depths and with a selection of different union materials, it has been found that the dyes listed below form a satisfactory basis on which a wide variety of colours may be obtained—

Caledon Printing Yellow 6G	C.I. Vat Yellow 27
Caledon Yellow 4G	C.I. Vat Yellow 13
Caledon Gold Orange 3G	C.I. Vat Orange 15
Caledon Brilliant Orange 6R	C.I. Vat Orange 3
Caledon Pink RL	C.I. Vat Red 43
Caledon Brilliant Red 3B	C.I. Vat Red 10
Caledon Red Violet 2RN	C.I. Vat Violet 14
Caledon Brilliant Violet R	C.I. Vat Violet 17
Caledon Dark Blue G	C.I. Vat Blue 16
Caledon Blue GCP	C.I. Vat Blue 14
Caledon Jade Green XN	C.I. Vat Green 1
Caledon Jade Green 3B	C.I. Vat Green 4
Caledon Green 7G	C.I. Vat Green 16
Caledon Olive Green B	C.I. Vat Green 3
Caledon Olive R	C.I. Vat Black 27
Caledon Brown R	C.I. Vat Brown 3
Caledon Dark Brown 6R	C.I. Vat Brown 38
Caledon Dark Brown 2G	C.I. Vat Brown 49

It is suggested that 75% of the caustic soda and hydrosulphite are employed in vatting the dye, the remainder being used for "sharpening" the dyebath and pretreating the material. The dye is vatted in a small volume at 120°F. for 15 min. prior to adding to the dyebath. The material to be dyed should be treated with 25% of the caustic soda and hydrosulphite at 40°C. (105°F.) for 10–15 min. before adding the dye, in order to obtain a uniform pH and to ensure that any oxidising substances have been removed from the system. The reduced vat dye is then added, and dyeing allowed to proceed at 105°F. for 45 min. An addition of 2% glue on the weight of material exerts a restraining effect on the adsorption of dye and has been reputed to act as protective agent for the wool. In the case of very pale colours, an addition of Dispersol VL can usefully be made with certain dyes. Again, with dyes normally applied to cotton by method 2, the normal amounts of common salt may be added to the dyebath in order to promote improved exhaustion.

Oxidation is carried out in the same manner as on cotton or viscose rayon with persulphate, percarbonate, or peroxide followed by acidification with dilute acetic acid and soaping with 2–3 parts of Lissapol NC per 1,000 parts of liquor at the boil for 20–30 min., preferably on the winch.

Azoic Dyes

Because the insoluble azoic dye is formed *in situ*, the wet-fastness properties of the resultant dyeing are high, and this class is therefore employed where maximum fastness is required. A wide variety of colours ranging from yellow to black is obtainable, but the principal use is in the production of bright, heavy reds, and this is the region in union dyeing where azoic combinations are potentially useful.

From the large number of different azoic pigments which can be produced by combining any coupling component with any diazo component, selection of the particular combination to be used is based in the first instance on the colour and fastness properties of the resultant dyeing, and secondly on the suitability of the combination for application under the proposed dyeing conditions. Essentially the dyeing process consists of four separate stages—

(1) Impregnation with coupling component solution
(2) Removal of excess coupling component
(3) Development with a selected diazo component
(4) Soaping of the resultant dyeing.

Compared with conventional union dyeing, this is a complicated process, and compared with dyeing on cellulose materials the selection of suitable combinations is complicated by the presence of the wool; difficulties can arise at any stage of the process. For these reasons, therefore, it is proposed to discuss in detail some of the factors which can profoundly affect the suitability of azoic combinations for dyeing wool–cellulose unions.

1. EFFECT OF CAUSTIC SODA CONCENTRATION

In the preparation of any coupling component solution it is necessary to add caustic soda in order to obtain dissolution of the coupling component and, in addition, an excess of caustic soda is necessary in order to achieve adequate stability of the solution. Wool dyers normally look askance at the treatment of wool with alkali, because of the damage likely to be caused to the protein fibre. However, with reference to coupling components where the dyeing is normally carried out cold, treatment of a 50 : 50 wool–cotton union yarn in 4 g. caustic soda (flakes) per litre cold for 1 hr. has virtually no effect on the tensile strength of the yarn (Table I). The minimum quantity of caustic soda recommended in dyeing cotton with Brenthols is 1·8 g./litre, and the effect of varying the concentration from this figure up to 4 g./litre on the solidity obtained on the union has been examined.

It has been found that, with any azoic combination, increasing amounts of caustic soda in the impregnating bath result in an increased uptake of coupling component by the wool. Dyeing on the wool with a caustic soda concentration of 4 g./litre is approx. 20% stronger than when the lowest caustic soda concentration is used. It will be clear from this that a combination which dyes one component much more heavily than the other cannot be made to dye solid by altering the caustic soda concentration. This effect can, however, be utilised in medium–heavy depths to effect an improvement with combinations of marginal solidity.

2. EFFECT OF DEPTH OF DYEING

With low concentrations of coupling component in the impregnation bath the colour on the wool is much yellower in all cases than when heavy depths are being dyed. Variations in either impregnating or developing conditions do not appear to influence this effect, and the yellowness is usually so great that it is impossible to effect correction by shading the wool with acid dye. Thus, in pale colours it is necessary to employ azoic combinations which reserve the wool and to fill-in the wool with acid dyes in a separate bath.

3. EFFECT OF TEMPERATURE ON SOLIDITY

Impregnation with coupling component solutions has been studied up to 40°C., and it has been found that in all cases, as the temperature is raised, the amount of coupling component adsorbed by the wool increases. At the same time, as adsorption by the wool increases, the amount taken up by the cellulosic fibre decreases. With Brenthols BC, BN, and BA the depth on the wool increases by 25% on raising the temperature from 15 to 40°C. The effect appears to be independent of depth, similar results being obtained with Brenthol concentrations of 0·25 and 2 g./litre. From the dyeings that have been produced, there appears to be no effect of temperature on the tone produced on the wool or on the yellowing of this fibre.

4. EFFECT OF USING BRENTAMINE FAST BASES OR SALTS ON DEVELOPMENT OF COLOUR

Experiments have been carried out in which 50 : 50 wool–cotton union cloth has been impregnated with a large number of different Brenthols at concentrations of 0·25, 0·5, 1, and 4 g./litre. Development has then been carried out with (*a*) diazotised Brentamine Fast Bases, (*b*) Brentamine Fast Salts. In pale depths (up to 0·5 g./litre) the same final colour is obtained on the wool or the cotton when using either the diazotised base or the salt.

In heavy depths there is a significant difference between the two compounds. In all cases the use of diazotised base gave a much heavier dyeing on the wool than that obtained with the corresponding Brentamine Fast Salt. It would appear that coupling occurs more rapidly and completely when the freshly prepared diazonium compound is used under these conditions, rather than the stabilised salt. This effect is most pronounced in the case of the production of a black from a combination of Brenthol GB (C.I. Azoic Coupling Component 13) with Brentamine Fast Red B (C.I. Azoic Diazo Component 5). The use of Brentamine Fast Red B Base results in the production of a black on the wool, whereas the use of Brentamine Fast Red B Salt produces only a dull yellowish green on the wool, and with the salt it has not been found possible to obtain the true black shade on the wool portion of a union. In these heavy dyeings increases in depth of up to 50% may be obtained on the wool by using certain Brentamine Fast Bases in place of the Brentamine Fast Salts.

On the cellulose portion of the union, on the other hand, there is no appreciable difference in depth of colour resulting from the use of base or salt.

5. EFFECT OF CONCENTRATION OF DIAZO COMPONENT

In the dyeing of azoic combinations on cellulose, it is well known that there is a definite combining ratio of a diazo component with any particular coupling component. Using this ratio together with a further factor related to the liquor ratio, it is an easy matter to calculate from absorption graphs the exact amount of base or salt required under any given conditions to couple with any given amount of coupling component. Dyeings have been carried out on 50 : 50 wool–cotton union material with coupling components at concentrations of 0·25–4·0 g./litre. Employing the absorption curves for cotton and the published combining ratios, development has then been carried out using the theoretical amounts of different diazo salts and bases. In addition, development has been carried out using $\frac{1}{4}$, $\frac{1}{2}$, 2, and 4 times the required amounts of salt or base.

Incomplete development occurs with $\frac{1}{4}$ and $\frac{1}{2}$ amounts of these compounds, and also increase in amount of diazo component up to 4 times the calculated amounts has virtually no effect on the depth of dyeing on either the wool or the cotton. The solidity between the wool and the cotton did not appear to be significantly affected by variations in developer concentration. Generally, development of the colour occurred very much more slowly on the wool than on the cotton.

6. EFFECT OF pH AND TIME ON DEVELOPMENT

Samples of the union material were impregnated with Brenthol solutions (2 g./litre). Developing baths using Brentamine Fast Salts were then prepared, and the pH adjusted in steps of 1 pH unit from 5 to 9 using sodium phosphate buffer solutions. Samples of Brenthol-impregnated material were then immersed in these solutions for the normal 30 min. and also for 1 hr. Within these limits, pH had virtually no effect on the solidity on the wool and the cotton, and doubling the time of development similarly did not affect the result. It was concluded, therefore, that, in spite of coupling being obviously slower on the wool than on the cotton, it was still complete within the normal development time of 30 min.

7. RECOMMENDED COMBINATIONS

From the results obtained in the tests outlined above it has been found possible to select a number of azoic combinations which give good solidity on 50 : 50 wool–cotton union materials (Table IV). Coupling components of high substantivity are considered to yield the most satisfactory results, both from the economic standpoint and also because good exhaustion is the most important factor in achieving dyeings of the highest fastness to rubbing. Where chlorinated wool is employed, there is a tendency for the wool to be dyed a little heavier than the cotton, but generally satisfactory results will be obtained.

TABLE IV

Azoic Combinations giving Good Solidity on 50:50 Wool–Cotton Union in Medium–Heavy Depths

BRENTHOL BC	C.I. AZOIC COUPLING COMPONENT 26
Brentamine Fast—	
Yellow GC Salt	C.I. Azoic Diazo Component 44
Orange GC Salt	C.I. Azoic Diazo Component 2
Scarlet GG Salt	C.I. Azoic Diazo Component 3
Scarlet G Base	C.I. Azoic Diazo Component 12
Red TR Salt	C.I. Azoic Diazo Component 11
Red KB Base	C.I. Azoic Diazo Component 32
BRENTHOL BN	C.I. AZOIC COUPLING COMPONENT 7
Brentamine Fast—	
Scarlet G Base	C.I. Azoic Diazo Component 12
Red KB Base	C.I. Azoic Diazo Component 32
Red TR Salt	C.I. Azoic Diazo Component 11
Red RC Salt	C.I. Azoic Diazo Component 10
Red AL Salt	C.I. Azoic Diazo Component 36
BRENTHOL AN	C.I. AZOIC COUPLING COMPONENT 4
Brentamine Fast—	
Orange GR Salt	C.I. Azoic Diazo Component 6
Scarlet G Base	C.I. Azoic Diazo Component 12
Red KB Base	C.I. Azoic Diazo Component 32
Red TR Salt	C.I. Azoic Diazo Component 11
BRENTHOL BA	C.I. AZOIC COUPLING COMPONENT 6
Brentamine Fast—	
Orange GC Salt	C.I. Azoic Diazo Component 2
Scarlet G Base	C.I. Azoic Diazo Component 12
Red KB Base	C.I. Azoic Diazo Component 32
Red TR Salt	C.I. Azoic Diazo Component 11
Red GL Salt	C.I. Azoic Diazo Component 8
BRENTHOL LTR	
Brentamine Fast—	
Scarlet G Base	C.I. Azoic Diazo Component 12
Scarlet R Salt	C.I. Azoic Diazo Component 13
Red TR Salt	C.I. Azoic Diazo Component 11
BRENTHOL CT	C.I. AZOIC COUPLING COMPONENT 8
Brentamine Fast—	
Orange GC Salt	C.I. Azoic Diazo Component 2
Orange GR Salt	C.I. Azoic Diazo Component 6
Scarlet G Base	C.I. Azoic Diazo Component 12
Red KB Base	C.I. Azoic Diazo Component 32
Red TR Salt	C.I. Azoic Diazo Component 11

The wet-fastness of these combinations is excellent, and the fastness to light is a minimum of 5 on the B.S.I. 1–8 scale with the exception of the combinations using Brentamine Fast Scarlet G Base and Brentamine Fast Scarlet R Salt, with which a pronounced blueing and dulling of the colour takes place on exposure to light.

8. RECOMMENDED DYEING PROCEDURE

For dyeing wool–cellulose union cloth with azoic combinations the preferred method of application is on the jig. Winch dyeing is unsatisfactory for the following reasons—

(*a*) The long liquor ratio results in poor adsorption of coupling component

(*b*) Running in rope form tends to introduce unlevelness in both impregnation and development

(*c*) Flotation of the material gives rise to unlevelness.

Whilst impregnation by padding is possible, the method falls down owing to the necessity for drying the material impregnated with alkaline coupling component solution before development, which results in undue degradation of the wool portion of the union.

Impregnation of the material with coupling component on the jig is preferably carried out for at least 20 min. in as short a liquor as conveniently possible in order to obtain the maximum adsorption of coupling component. An addition of common salt (20 g./litre) can also be made with Brenthols BC, AN, BA, LTR, CT, BN (10 g./litre) in order to improve the exhaustion.

After impregnation and before development, excess, loosely held coupling component must be removed if maximum rubbing fastness is to be obtained. This is best done in the case of wool–cotton unions by giving a squeeze, but if this is impracticable the next best alternative is a brine rinse in a cold solution containing 50–100 g. common salt and 1·5 g. caustic soda per litre for 10–15 min.

Development is then carried out on the jig by setting the bath with the required amount of diazo component with the addition of common salt (25 g./litre) to prevent bleeding of coupling component into the liquor during the early stages of development. Development should be carried out for a minimum of 20 min., and normally 30 min. should be allowed.

The material is then acidified by running in a bath containing hydrochloric acid (3 ml./litre) at 40°c. for 10–15 min. to remove mechanically held compounds formed during development, and rinsed in clean, cold water for 5 min.

The process is completed by soaping the material with Lissapol N (3 g./litre) at the boil for 15 min. This should be carried out on the winch or even on the dolly in order to obtain relaxation of the material and to obtain the maximum mechanical movement of the material so as to facilitate efficient removal of loose colour. Finally the material is given a warm-water rinse.

The dyeing of wool–cellulose union yarns is preferably carried out in package form in circulating machines. Care is required in winding the cheeses to ensure even density throughout the package, and soft ends are desirable. All solutions should be sieved into the dyebath in order to remove suspended material which could result in poor rubbing fastness. After impregnation and before development, excess coupling component solution must be removed either by hydro-extraction or by suction. A brine rinse may be used as an alternative, but is not as efficient in obtaining good rubbing fastness as the two methods previously mentioned. Development is carried out cold for 30 min. in the same way as for piece goods, but an addition of Azopol A (2 g./litre) to the bath helps to prevent any insoluble pigment formed in the liquor from being filtered on to the outside of the packages. The acidification and soaping treatments are similar to those used in piece dyeing, but, since there is no movement of the material in this form, removal of loose pigment cannot be as efficient as in the case of piece goods, and increased looseness to rubbing must be

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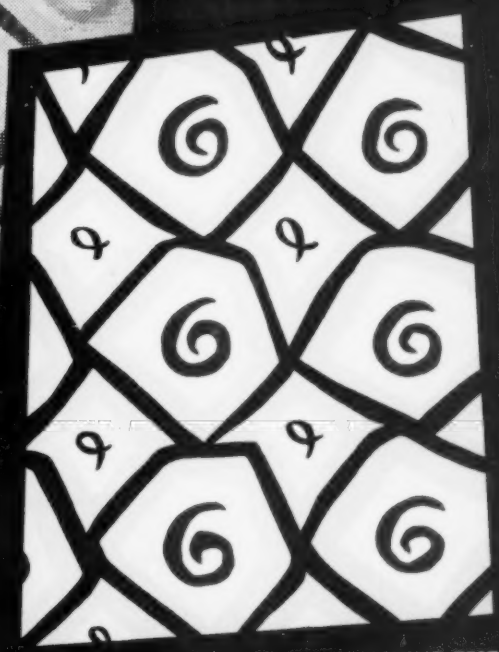

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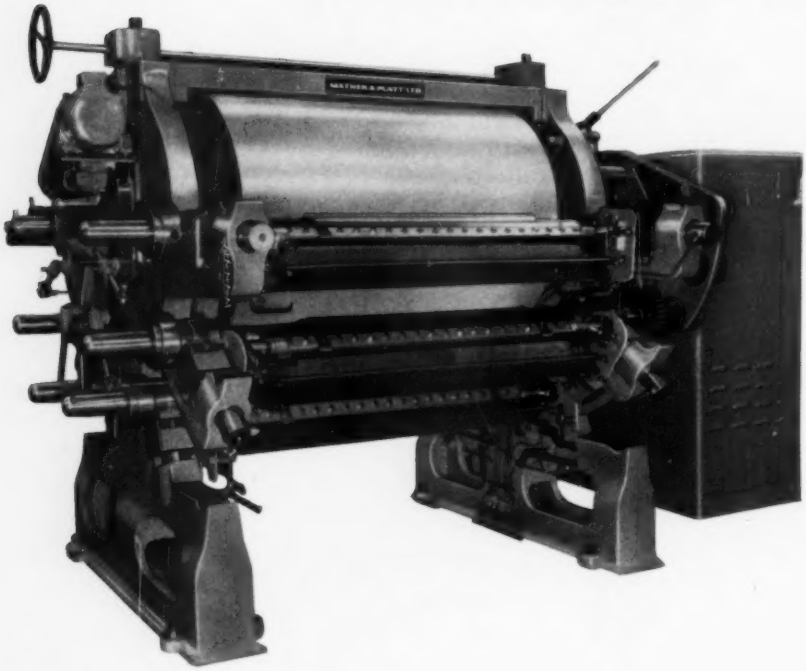

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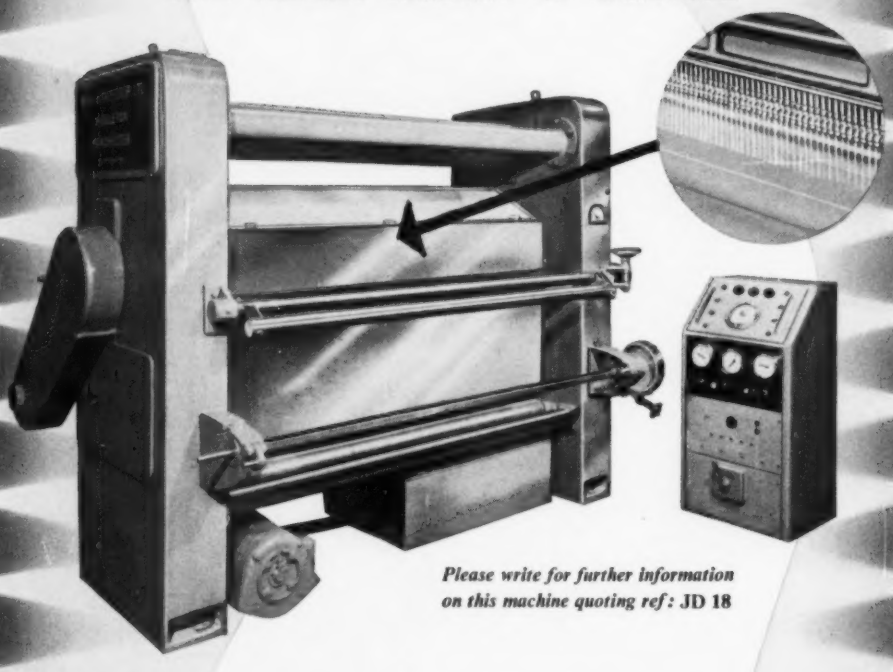

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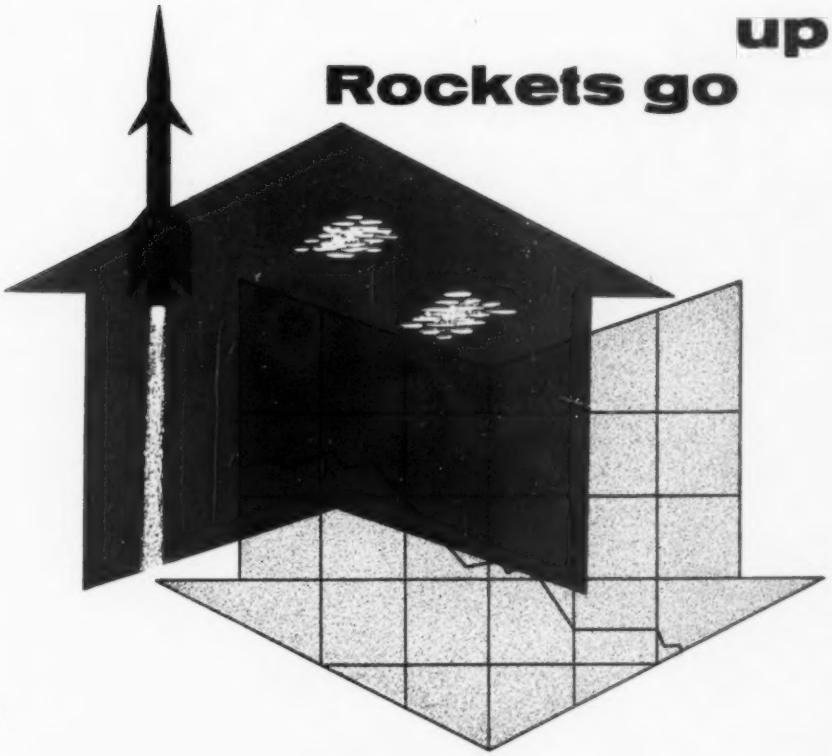

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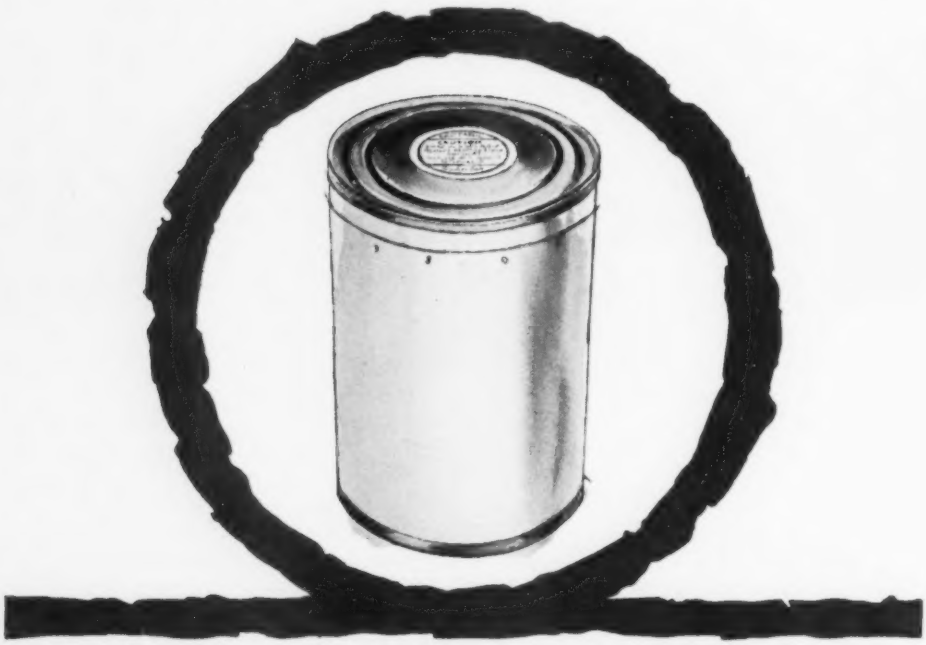

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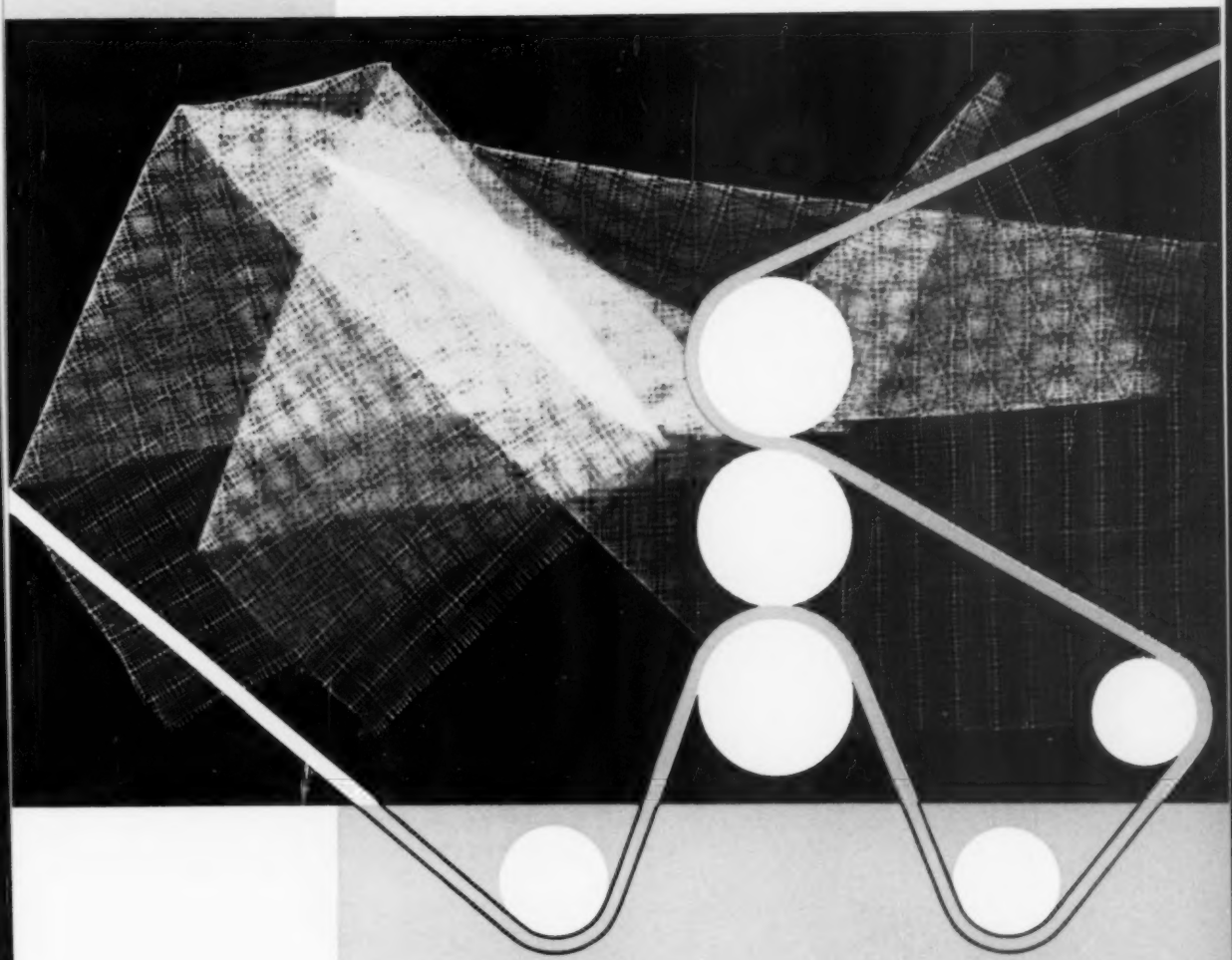

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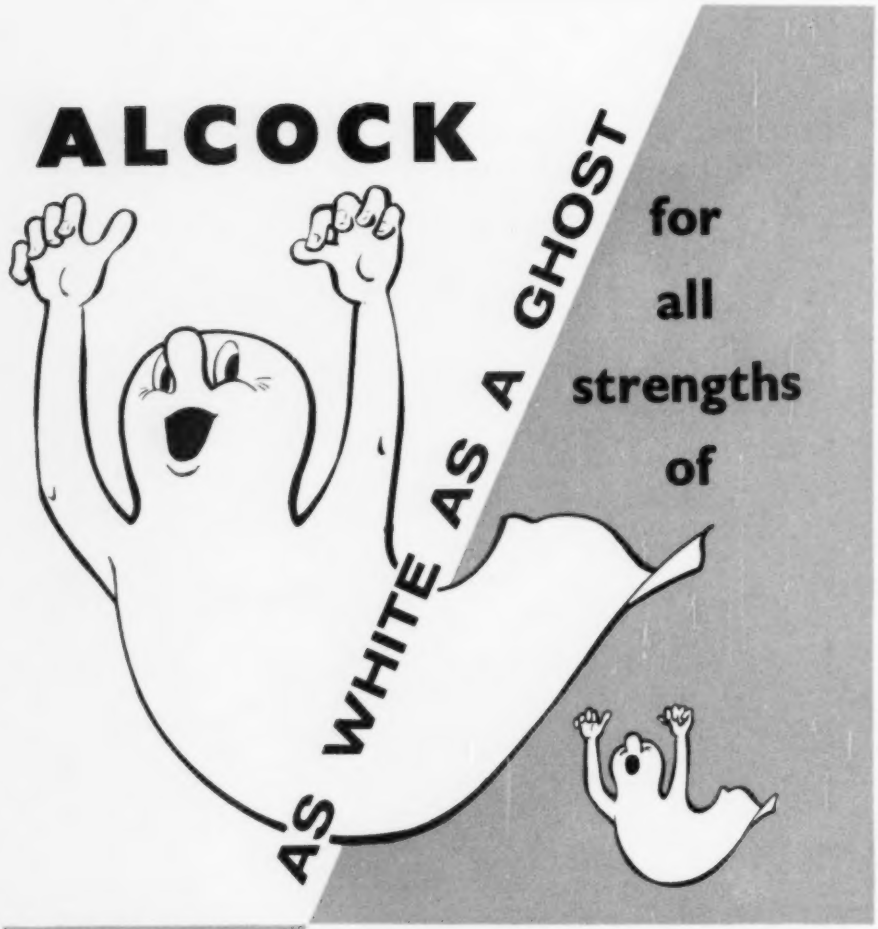

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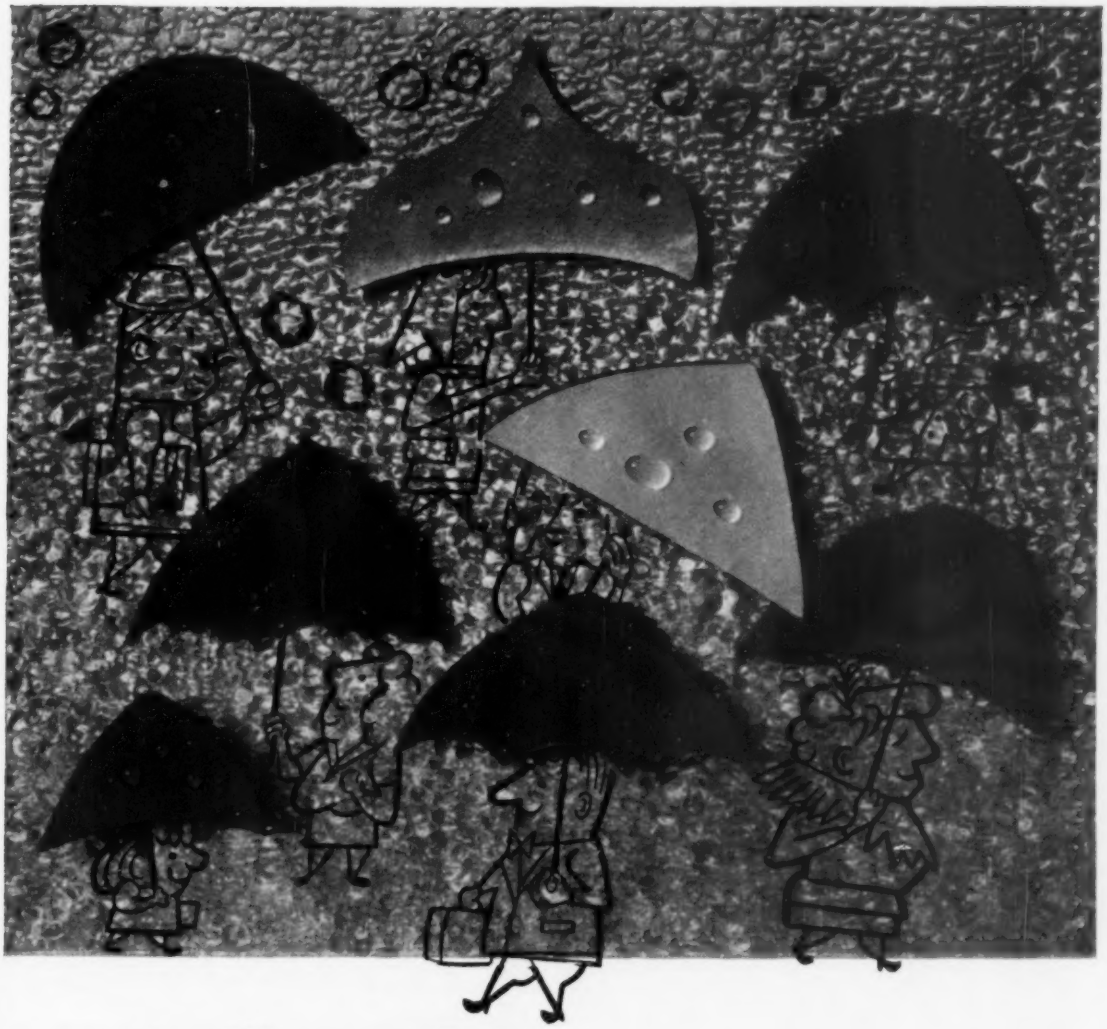

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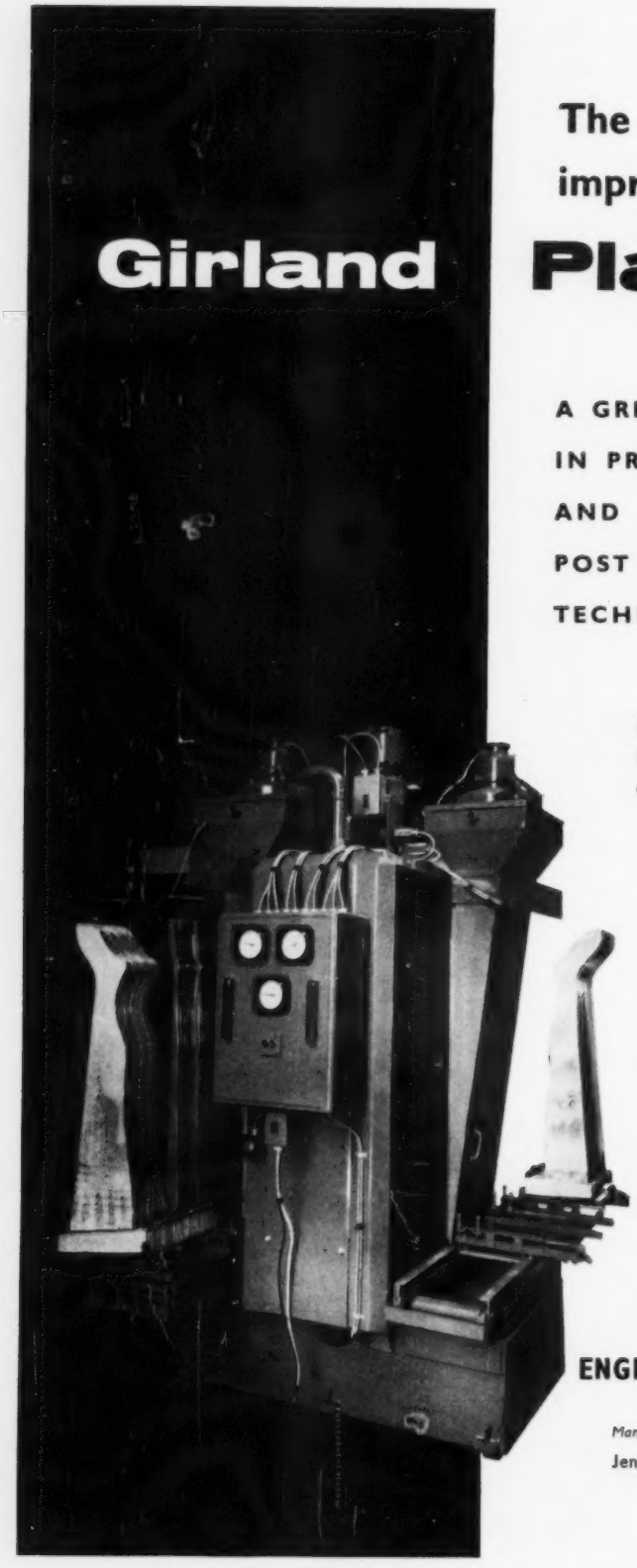

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Shade

Neutral Grey.

Main features

C.II class dyestuffs.
Cover dead cotton and viscose rayon of varying affinity.

Fastness Properties

Fast to weathering.
High fastness to light.
Very good all-round fastness.
High fastness to chlorine and peroxide bleaching.
Good fastness to sodium chlorite bleaching.
Fast to mercerising.
Resistant to synthetic resin finishes.

Uses

For styles requiring maximum fastness – both yarn and piece goods.
Specially suitable for shirtings on account of fastness to peroxide.
The Micro Disperse brand should be used for pigmenting and continuous dyeing.

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Sole Concessionaire in the United Kingdom

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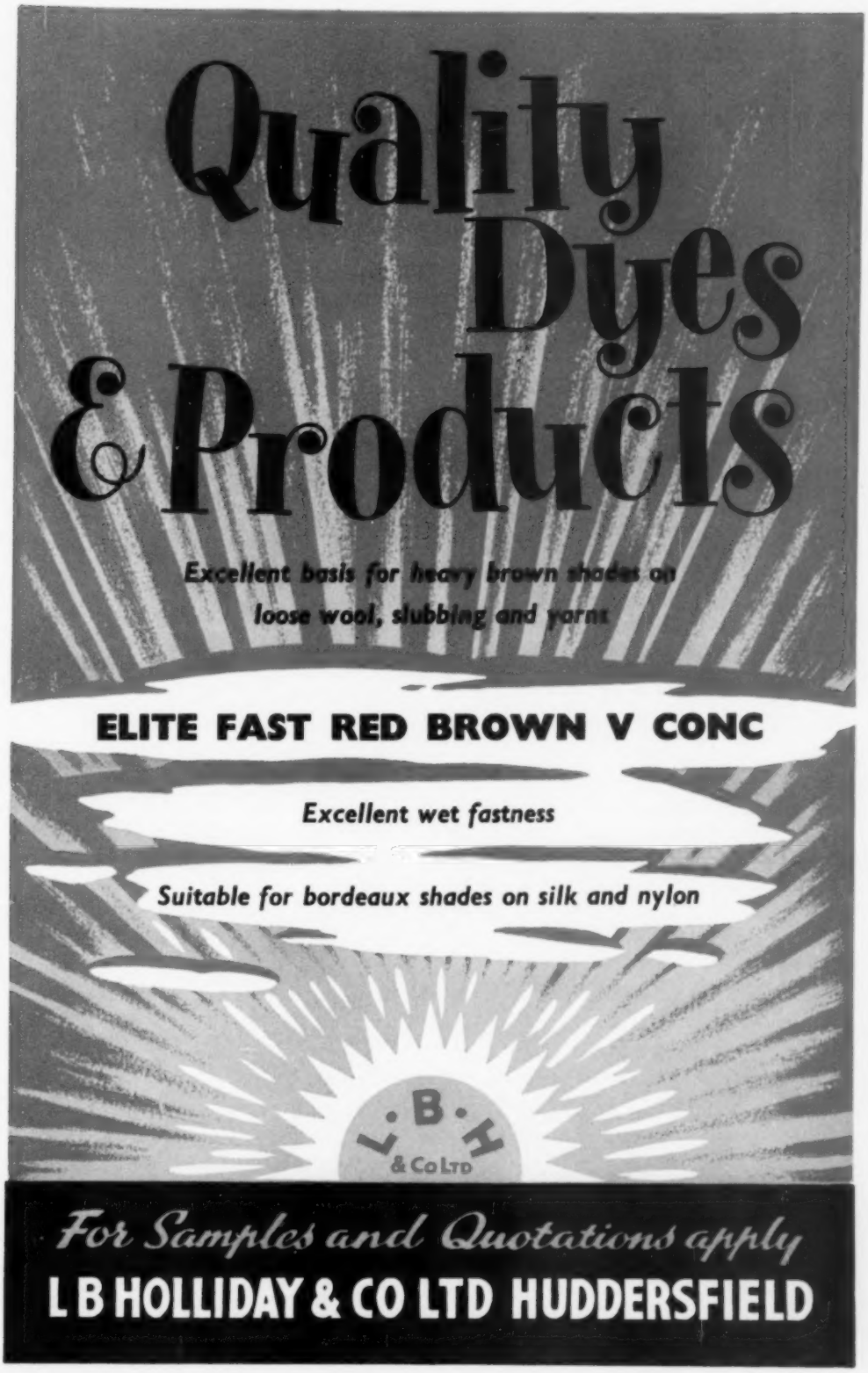
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ELITE FAST RED BROWN V CONC
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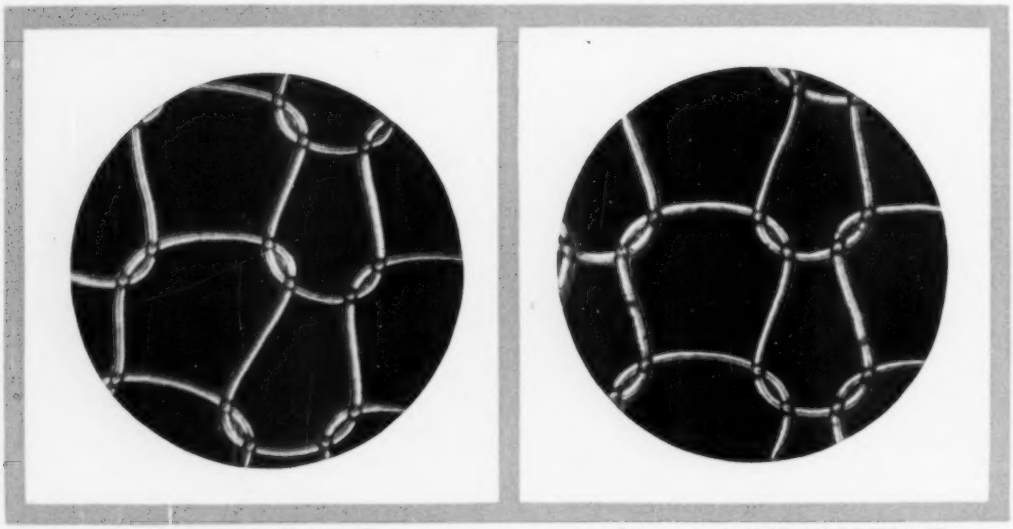
FOR NYLON HOSIERY FINISHING



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A specially developed range of VINAMUL Emulsions based on Vinyl Acetate and Methacrylate Polymers is available for the finishing of nylon hose to meet the varying requirements of handle and snag resistance which are demanded by the hosiery finisher. The range includes both cationic and anionic types for application by exhaustion and standing bath processes and typical grades are:

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®Remazol
Dyestuffs

These reactive dyes have been developed for dyeing and printing of cotton, and of viscose rayon staple and filament. They have wide fields of application. REMAZOL dyestuffs are readily soluble and unaffected by hard water.
Penetration and level dyeing properties are excellent, and for shading they may be added to the dyebath during and after development. Dyeings and prints from REMAZOL dyestuffs are fast to rubbing and cross-dyeing. They are suitable for permanent finishes, PVC coating, dry-cleaning treatments, and are resistant to plasticizers.

At present the range comprises the following brands:

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Remazol Red B
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Remazol Red Violet R
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Distributors in Eire for Dyestuffs and Auxiliaries:
JOHN McWADE & Co. Ltd., 11, Nassau Street, Dublin

expected. This can be corrected in subsequent piece scouring after the yarn has been converted into fabric.

Preparation of the coupling component and developer solutions is effected in the same way as for the dyeing of cotton or viscose rayon.

Conclusions

From the work described, it will be seen that it is possible to obtain dyeings on wool–cellulose unions possessing wet-fastness properties not previously considered practicable. Whilst the dyeing methods are more complicated than those normally associated with conventional union dyeing, they are nevertheless practicable provided that suitable dyeing equipment is available. It has been shown that the production of solid colours depends on the condition of the individual fibres in the blend and the choice of suitable dyeing conditions and dyes, and careful attention to all these factors is essential if satisfactory results are to be obtained. By the methods outlined, it is possible to apply reactive, onium, vat, and azoic dyes to wool–cellulose unions to obtain any desired colour, and this should help in raising the status of wool–cellulose unions in the textile field.

Dyehouse Laboratories
Imperial Chemical Industries Ltd.
Hexagon House
Manchester 9

(*MS. received 17th April 1959*)

CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Annual Golf Competition

Most readers, particularly golfers, will know that the Goodall Trophy is played for annually at a venue in the North-western area. I have competed only for the past two years, but on both occasions I must admit that for such a magnificent trophy the entries have been well below what one would expect.

Taking into consideration that many members who would like to compete are prevented from doing so by having to travel long distances, I feel that a greater effort could be made by members who live within a radius of fifty miles from Manchester.

A great deal of work is entailed in organising this event, and full credit must go to Mr. W. R. Leigh for his efforts in making what is a very successful and entertaining day for the competitors.

I feel that perhaps insufficient publicity may be one reason for the poor support, but possibly other members have suggestions which may provide better support in the future, and no doubt Mr. Leigh would be pleased to receive them.

G. V. W. Kendell

118 Victoria Avenue East
Manchester 9

17th June 1959

Notes

Meetings of Council and Committees

July

Council— 15th

Publications— 14th

International Federation 1959 Congress— Planning and Co-ordinating— 10th

Data on Identification of Dyes on Textile Fibres— 3rd

Colour Index Editorial Board— 10th

Textbooks and Monographs Subcommittee— 14th

Review of Textile Progress— 24th

Election of Fellows

At the meeting of Council held on 6th May 1959 the following were elected Fellows of the Society—

Herbert Carlisle
(*London; Senior Dyeing Technician with Chemstrand Ltd.*)

Herbert Hilton
(*Stockport, Cheshire; Technical Director, J. J. Hadfield Ltd.*)

Deaths

We regret to report the death of Dr. W. Flügel and Mr. H. Lamb.

Seventh London Lecture

The title of the seventh London Lecture is *Colour and Design in Wallpapers*, and the lecturer will be Mr. Eric A. Entwisle, a director of The Wall Paper Manufacturers Ltd. The lecture is to be held at the Waldorf Hotel, Aldwych, London W.C.2, on Friday, 23rd October 1959, at 7 p.m., and will be followed by a dinner.

Annual Golf Competition

The Annual Golf Competition for the Goodall Trophy and other prizes took place at Leek Golf Club on Sunday, 14th June 1959, in glorious weather. The Trophy was won by Mr. R. Jessop with a score of 92 − 22 = 70. Three members tied for second place with a net score of 75: Mr. G. V. W. Kendell (80 − 5 = 75) took the scratch prize, presented by Mr. J. W. Reidy; on the spin of a coin Mr. F. Barrett (91 − 16 = 75) took second prize, presented by Mr. Kendell, and Mr. J. Broadhurst (84 − 9 = 75) took third prize, presented by Mr. W. R. Leigh.

West of England and South Wales Section

Inauguration of New Section of the Society

A West of England and South Wales Section of the Society of Dyers and Colourists was inaugurated on 26th June 1959, at a meeting and dinner held at the Grand Hotel, Broad Street, Bristol. Mr. John Boulton (*President of the Society*) took the Chair supported by Mr. F. Malcolm Stevenson (*President-elect*), Mr. R. Woods and Mr. H. W. Taylor (*Chairman and Honorary Secretary of the London Section*), Mr. J. W. Nicholls (*General Secretary*), and Dr. C. J. W. Hooper (*Editor and Technical Officer*). Good wishes and apologies for absence were received from Mr. L. Morton Wood and Mr. A. Waddington (*Honorary Secretary and Honorary Treasurer of the Society*), and from Mr. Clifford Paine (*Immediate Past President*). The meeting was well attended by close on 100 textile dyers and colourists together with colourists from other trades such as leather and paper, and also members of dye-manufacturing firms.

After referring to his pleasure that a new Section of the Society should be formed during his period of office as President, Mr. Boulton said that he was surprised, having in mind the activities carried on in that part of the country, that no Section of the Society had been formed there previously. The surname *Dyer* was first recorded, from 1429 to 1453, for such persons as Henry of Witney, Thomas of Corscombe, William of Fordenbridge, Adam of Chipping Campden, and John of Warminster, who started as dyers in that area and whose names ultimately became *Dyer*, *Dier*, or *Diere*. There was a great deal of activity in that part of the country concerned not only with textile dyeing but with the leather and other industries served by the Society. General topics which could come under the heading of textile chemistry, for instance, were of interest in the production of bonded-fibre fabrics and regenerated cellulose films.

One problem was that these industries were fairly widely spread, and they had to decide whether, in spite of this wide dissemination, they could form a co-operative Section of the Society which could look forward to meeting regularly and in sufficient numbers to make it worth while. Council had approved the formation of a Section in the West of England, but had taken no decision whether it should embrace South Wales or whether there should be two Sections. So they had to attempt to implement Council's decision to form a Section in the area, and then to decide whether there should be one or two Sections.

Mr. Stevenson said that he was struck by the very substantial support that had already been given to the thought of forming a Section in the area, and was tremendously impressed by the attendance. He suggested that they might consider holding meetings alternately in different towns, as was the practice of the Midlands Section. Mr. Stevenson also expressed appreciation of the tremendous amount of preliminary work done by the General Secretary and of firms who gave staff facilities for attending meetings.

Mr. Nicholls urged that joint meetings should be held with local sections of the Textile Institute, the Chemical Society, and other similar organisations, and advocated the issuing of a combined programme of all scientific and technical meetings to be held in the area. He stated that there was a potential membership in the West of England area of one hundred. They already had a basis of 31 members, with more on the way. The membership was widely spread, generally with an average of 3–4 members in places such as Bridgwater, Tiverton, Stroud, Taunton, Yeovil, Ilminster, Exeter, Plymouth, and elsewhere. Although not so confident about South Wales, he felt that they could look forward comfortably to something in excess of 50 members: there was a basis of 21 members, and others had indicated interest.

The President said that they should first proceed with the formation of a West of England Section, and then decide whether or not to make any recommendation in regard to a separate Section in South Wales. Boundaries could be fixed later on the basis of experience. Mr. Taylor stated that the London Section had long been conscious of the fact that in the West of England there were members of the Society they could not cater for, and therefore the formation of a West of England Section had had the blessing of the London Section from the start.

Eight members were present from South Wales, and Mr. G. K. Mecklenburgh said that he did not feel at the moment that a separate South Wales Section was justified. Thirteen of the 21 members were, in fact, from one firm and worked very closely together. Members in South Wales who might live at some distance from Cardiff or Newport might find attendance at lectures difficult because of the poor train connections.

The President then asked the meeting to consider a resolution that "this meeting decides to set up a Section of the Society of Dyers and Colourists serving the West of England, with which is included at present South Wales". The resolution, proposed by Mr. B. N. Symonds and seconded by Mr. F. Courtney Harwood, was carried unanimously. Mr. Symonds suggested that the new Section could hold, perhaps, two meetings centred on Bristol, one in South Wales, and one in South-west England, e.g. at Taunton. Mr. Symonds also emphasised the need for employers' co-operation in allowing people to reach these centres sufficiently early in the evening to attend the meetings.

The President said that it would be wise to elect a provisional Committee with a Convener or Honorary Secretary, and the President-elect thought that a balanced Committee was needed, with representatives of each area— South-west England, South Wales, Bristol, Gloucester, and Stroud.

On the suggestion of Mr. Woods, it was proposed and seconded that Mr. J. N. Littler be Convener, and Mr. Littler accepted. The following Committee, proposed by various members present, then

accepted office—Mr. E. Holden, Mr. S. Shaw, Dr. G. Welford, Mr. Dick Carter, Mr. F. Jamieson, Mr. J. W. Young, Mr. M. Magnew, Mr. R. Beaumont, Mr. J. F. D. Butterworth, Dr. Trevor Smith, and Mr. A. S. Levesley. The Committee was empowered to co-opt additional members.

In the general discussion that followed, Mr. Stevenson said that the Section would want to get into its stride for the winter session, and it would be useful for headquarters to know their thoughts as to the type of lectures that would be acceptable in the area. Dr. Hooper offered to forward copies of the list of lecture titles already circulated to the other Sections, which had quite a wide coverage of both textile and non-textile topics. Mr. Woods said that, although no Section dealt with more varied industries than the London Section, they had found it possible to satisfy most tastes. It was not important that lectures should be on the subject nearest to their hearts or their work. The most important thing about the lectures was that they did bring dyers and colourists together.

Mr. Symonds referred to the honour of having the President with them and thanked him for coming to the West of England to inaugurate this new Section, which they all trusted would flourish.

The President, in reply, expressed the hope that it would be his privilege to take the Chair at the first meeting of the Section next winter. He congratulated Mr. Littler and the Committee on having made this start, and promised them that they would have all possible support from the Society's headquarters.

At the first meeting of the Section Committee, held on July 2nd, Dr. G. Welford was elected Chairman. The full list of Committee members and officers was published in the July issue of the *Journal* (**75**, 347).

Recent Developments in the Chemistry of Natural and Man-made Fibre-forming Polymers

A special short course on the above subject will be held on 30th and 31st October 1959 in the Department of Chemical Technology of the Bradford Institute of Technology. The fee for the course is £1 10s. 0d. Arrangements will be made for informal discussions and visits to the Polymer Research Laboratories of the Institute during the evening of Friday, October 30th. Further information can be obtained from the Registrar, Institute of Technology, Bradford 7, Yorkshire.

New Books and Publications

Peter Griess

Leben und Wirken eines grossen Farbstoffchemikers

By August Wingler. Leverkusen-Bayerwerk: Farbenfabriken Bayer AG. 1958. Pp. 39. No price.

This small but attractive publication by Farbenfabriken Bayer is the firm's own centenary tribute to Peter Griess, with whom there were tenuous links through Böttinger and Duisberg.

Whilst disclaiming any intention of embarking on an exhaustive account of Griess's life and work or of recording anything new, the author has very successfully winnowed the salient facts from the greatly longer accounts of Hofmann and Caro. He brings the story to modern times by sketching the most important developments in the chemistry of azo dyes since the discovery of Chrysoidine in 1876 infused new life into a dyestuffs industry which by then tended to be overshadowed by Alizarin and derivatives of Magenta. For those with an appetite for detail, there is a list of 48 references.

Dr. Wingler shares a common belief that the Royal College of Chemistry in London was the birthplace of Perkin's Mauve, the first of the synthetic dyes. In fact, the discovery was made in Perkin's makeshift home laboratory and in 1856, not 1858 as stated. Apart from this minor inaccuracy, Dr. Wingler's account is unexceptionable.

Not the least interesting feature of this publication is the series of 18 illustrations, including three in colour, some of which are made public for the first time.

W. H. Cliffe

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index*

I—PLANT; MACHINERY; BUILDINGS

PATENTS

Carbon Black Furnace

Godfrey L. Cabot *BP* 814,095

A method of premixing the air and fuel gas in the furnace throat without causing vibration and pulsation. It enables closer control of the production of the pigment. C.O.C.

Densification of Carbon Blacks (C.I. Pigment Black 6 and 7)

Godfrey L. Cabot *BP* 814,101

A horizontal rotating drum contains a roller having two "legs" i.e. longitudinally spaced eccentric projections in the same radial plane. The roller is as long as the drum but is not attached to it and so is free to roll as the drum rotates. With each revolution of the roller it is lifted by the eccentric legs and then dropped back on to the pigment on the drum. Ink grade Carbon Black wetted with 2–12% of water either before or after being placed in the drum is continuously added to one end of the rotating drum to a depth not above the diameter of the roller in its raised position, the densified pigment being continuously withdrawn at the other end. C.O.C.

Wet Processing of Cloth in Open width

DuP *USP* 2,869,976

A star frame on which the cloth is loosely wound and whose bottom position is immersed in the treating liquor.

It is so made that as it rotates the gravitationally induced motion of the bars supporting the cloth move the cloth round the frame so that uniform contact between the cloth and the liquor is obtained over an appreciable number of rotations of the frame. C.O.C.

Cleaning and Coating Sized Glass Fibre Fabric
Owens-Corning Fiberglas Corpn. *USP* 2,868,669

Apparatus for the heat cleaning, setting and improving the draping qualities of glass fibre fabric in which the fabric is heated to 500–900°F. immediately before it enters a combustion chamber where it is heated to 900–1300°F. The preliminary heating drives off enough of the size to enable the complete removal of the remainder of the size in the combustion chamber to be carefully controlled. C.O.C.

Calico Printing Machine
Cranston Print Works Co. *USP* 2,870,705

A machine in which the register, both across and along the cloth, is quickly, easily and precisely set and, once set, is maintained over long periods without attention. The machine is readily stopped without tearing the cloth. It has a duct or blade drive of simple construction and a wide range of adjustment so that the duct or blades may be reciprocated with both simple and complex motions. The print roll drive and the registering device are combined in one simple mechanism together with means allowing the print roll to move radially of the backing drive. It is unnecessary to disassemble much of the machine or to lift the backing cylinder in order to replace the endless backing cylinder blanket. C.O.C.

Paper Coating Machine
Black-Clawson Co. *USP* 2,870,738

Apparatus which can be quickly and easily changed from direct coating to reverse coating and vice versa by simple rearrangement of the rolls carried by the main frame and without affecting the main direction of travel of the web. C.O.C.

Spreading Liquid or Plastic on Travelling Cloth or Paper
Industrial Insulations *BP* 814,100

Modification of *BP* 681,278 (J.S.D.C., **69**, 31 (1953)). The cloth is led through the machine so that it has a run in which one side of the cloth is uppermost followed after a change in direction by another run in which the other side of the cloth is uppermost. Two spreading units are arranged so that a coating is applied to the cloth on each of these runs. C.O.C.

Continuous Diazotisation Process (IV p. 434)
Storage of Backgreys for Roller Printing (IX p. 438)

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Sodium Nitrite as a Corrosion Inhibitor
S. Sussman, O. Nowakowski, and J. J. Constantino
Ind. Eng. Chem., **51**, 581–584 (April 1959)

Textile Chemicals and Auxiliaries
Silk and Rayon, **32**, 958–959, 966 (Sept.); 1113–1114, 1133 (Oct.); 1223–1224, 1228 (Nov.); 1329, 1333–1334 (Dec. 1958); **33**, 43–44, 50 (Jan.); 157–158, 162 (Feb. 1959)

PATENTS

Emulsions of Hydrocarbon Polymers
Esso Research & Engineering Co. *USP* 2,871,137

Emulsions of unusual mechanical and chemical stability are produced by treating a hydrocarbon polymer to convert some of it to an emulsion which will emulsify the remainder of the polymer, e.g. a hydrocarbon polymer containing at least a little unsaturation is treated with maleic, chloromaleic or citraconic anhydride and the adduct obtained is neutralised with a basic compound of an alkali metal. The resulting salt has surfactant characteristics and is particularly effective as an emulsifying agent for either the polymer from which it was made or any other saturated or unsaturated hydrocarbon polymer. C.O.C.

Sequestering Agents for Polyvalent Heavy Metal Ions
Atlas Powder Co. *USP* 2,868,724

Compounds of formula—

$$(C_6H_{13}O_5)\text{–}N\text{–}\begin{bmatrix} \text{–}R_a \\ \text{–}(C_nH_{2n}COOY)_b \\ \text{–}\left(\begin{matrix}\text{CHCOOY}\\ |\\ \text{CH}_2\text{COOY}\end{matrix}\right)_c \\ \text{–}CH\!<\!\begin{matrix}\text{COOY}\\ \text{COOY}_d\end{matrix} \end{bmatrix}$$

($C_6H_{13}O_5$ = hexanepentol residue of a hexityl amine; R = H, Alk of 1–3 C or low hydroxyalkyl of 2 or 3 C; Y = H or monovalent alkali metal; n = 1–2; a = 0–1; b = 0–2; c = 0–1; d = 0–1; $a + b + c + d$ = 2), e.g. *N*-methyl(methyl)*N*-carboxymethyl glucamine, *N*-methyl *N*-glucyl aspartic acid, *NN*-bis carboxymethyl glucamine and *N*-methyl *N*-carboxymethyl fructamine, are excellent sequestering agents for the ions of polyvalent metals of atomic number > 24, e.g. Fe, Cu, Mn, Ni and Pb. C.O.C.

Aluminium Soap Water-repellent Compositions
Nopco Chemical Co. *USP* 2,868,666

When a water-soluble soap and the salt or free acid of a water-soluble phosphorus-containing wetting agent are coprecipitated by aluminium, the product is insoluble in water but readily soluble to high concentrations in organic solvents to yield stable solutions of good quality and freely flowable. Thus a mixture of sodium stearate and the 2-ethylhexyl ester of pentasodium tripolyphosphate dissolved in water and precipitated with aluminium at 62°C. yields a stable 30% solution in perchloroethylene. C.O.C.

Organosilicon–Titanium Compositions as Water-repellent Finishes
Dow Corning Corpn. *USP* 2,868,750

A mixture of a benzene-soluble organopolysiloxane, or benzene-soluble titanic ester of an aliphatic alcohol of < 20 C, an organic solvent and a nitroalkane or nitrobenzene gives a good water-repellent finish to fibrous materials, particularly leather. C.O.C.

Cyclopentadienyl Transition Element Catalysts for Silicone Finishes
Union Carbide Corpn. *USP* 2,868,751

Compounds of formula $R_nMX^1{}_mX^2{}_n$ (R = subst. or unsubst. cyclopentadienyl; M = transition element; X^1 and X^2 = same or different, Cl, Br or I; m = 0 or 1; n = 0, 1 or 2) are superior catalysts for curing organic solvent-soluble silicones. They are effective at room temperature. Examples are bis-cyclopentadienyl iron, bis-cyclopentadienyl titanium bromide chloride, and bis-cyclopentadienyl zirconium dichloride. C.O.C.

Sulphomethylcelluloses
Mo och Domsjo *BP* 813,900

Depending on the degree of substitution (average number of sulphomethyl groups per anhydroglucose unit) sulphomethylcellulose is insoluble or more or less soluble in water. At substitution < 0·1 the sulphomethylcellulose is insoluble, at > 0·1 substitution it becomes soluble the solubility increasing with increase in substitution. Sulphomethylcelluloses have wide uses as thickening, binding, dispersing, and emulsifying agents. C.O.C.

Dry Size for Paper
A. Schneider *BP* 813,739

A dry rosin size is made by mixing rosin with a liquid hydrocarbon (0·5–5·0% on wt. of rosin) and Na aluminate (15–30%) with or without simultaneous or subsequent heating and/or addition of water to be later evaporated off. It is completely soluble in cold water. When added dry to paper pulp it is quickly distributed throughout the pulp and precipitates in neutral medium. C.O.C.

Hardening of Gelatin with 2,3-Dihydroxydioxan
Eastman Kodak Co. *USP* 2,870,013

Gelatin containing 0·2–5·0% of 2,3-dihydroxy-1,4-dioxan is resistant to either cold or hot water. C.O.C.

Flame Resistant Textiles (X p. 440)

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Replacement of Sulpho Group by the Hydroxyl Group in Aromatic Compounds
III—Kinetics of the Reaction of Sodium Salts of 2,6- and 2,7-Naphtholsulphonic Acids with Sodium Hydroxide
S. M. Shein and N. N. Vorozhtsov
Ukrain. khim. zhur., **24**, (4), 643–647 (1958)

Investigation of the above has shown them to be third order reactions, the dependence of the rate constants on temp. being represented by—

$$\log k = 9{\cdot}65 - \frac{8{,}400}{T} \text{ (2,6 isomer)}$$

$$\log k = 10{\cdot}8 - \frac{8{,}700}{T} \text{ (2,7 isomer)}$$

G.J.K.

Technology of Food Colorants
B. R. J. Thomas *Chem. and Ind.*, 468–470 (11 April 1959)

A discussion of the problems confronting the manufacturer and user of food colorants. Properties of an ideal colorant are given, and the colorants given an A designation in the U.K. permitted list are examined. The difficulties of blending, to obtain colours not in the list, and the use of natural dyes are also mentioned. Finally the peculiar differences between the regulations in different countries and the consequent problems to the food exporter are explained. P.B.S.

Biochemical and Pharmacological Aspects of Food Colorants and Flavours
R. T. Williams *Chem. and Ind.*, 473–477 (11 April 1959)

A brief summary of the legal position with respect to food additives, in both the U.K. and the U.S.A., is first given, then the information ideally needed on such additives. The biochemical and pharmacological activity of the sulpho and azo groups is described. The absorption of sulphonic acids by the intestine and their cathartic and carcinogenic activity are shown by examples from the triarylmethane and azo classes. There is a warning that, whilst the presence of sulpho groups reduces carcinogenicity, this is not always the case. The enzymatic splitting of the azo group, giving two amines, is described by reference to those dyes capable of giving rise to naphthylamines. Many of these have carcinogenic properties, and the detoxicating effect of added sulpho groups is again illustrated. 47 references are given. P.B.S.

Steric Factors in the Properties of Dyes containing a Diphenyl Nucleus. XI—Azo Dyes derived from Diphenyl, *p*-Terphenyl, and *p*-Quaterphenyl
B. M. Krasovitskii and N. I. Titarenko
Ukrain. khim. zhur., **24**, (4), 481–486 (1958)

A comparative investigation of colour and affinity for cotton of mono- and bis-azo dyes derived from diphenyl, *p*-terphenyl, and *p*-quaterphenyl has been carried out. Accumulation of benzene rings between the azo groups of these dyes causes a hypsochromic shift of the absorption maxima and increases the affinity for cotton. G.J.K.

Internuclear Cyclisation. XIII—Decomposition of Diazonium Salts prepared from *N*-*o*-Aminobenzoyldiphenylamines. A New Molecular Rearrangement
D. H. Hey and T. M. Moynehan
J.C.S., 1563–1572 (April 1959)

Thermal decomp. of an aq. soln. of the diazonium chloride from *N*-*o*-aminobenzoyldiphenylamine gives 10-phenylphenanthridone and *N*-*o*-hydroxybenzoyldiphenylamine, but catalytic decomp. at room temp. by the addition of copper powder gives 10-phenyl-phenanthridone and diphenyl-2-carboxyanilide. Evidence on the nature of the anilide-forming rearrangement follows from the data of similar reactions with the *N*-*o*-aminobenzoyl deriv. of di-*p*-tolylamine, 4-methyl- and 4-chlorodiphenylamine. The rearrangement occurs only in the catalytic decomp. of the diazonium salt. H.H.H.

Photoreduction of Methylene Blue (C.I. Basic Blue 9) on Titanium Dioxide
R. Ya. Mushii and A. V. Pamfilov
Ukrain. khim. zhur., **24**, (4), 462–466 (1958)

An increase in the concn. of ferric and cupric ions at the surface of TiO_2 (anatase) lowers the photocatalytic activity of TiO_2, whilst tungstate ions produce the reverse effect. The authors apply the redox mechanism suggested by Weeyl and Förland (*Ind. Eng. Chem.*, **42**, 257 (1950)). G.J.K.

Magenta—Milestone in Dyeing History
M. Schofield *Dyer*, **121**, 853–855 (22 May 1959)

Metabolism of Sulphobromophthalein
G. M. Grodsky, J. V. Carbone, and R. Fanska
Nature, **183**, 469–470 (14 Feb. 1959)

Photoreduction of Dyes in Rigid Media. I—Triphenylmethane Dyes
G. Oster, J. Joussot-Dubien, and B. Broyde
J. Amer. Chem. Soc., **81**, 1869–1872 (20 April 1959)

Some triphenylmethane dyes are unaffected by light when they are in aqueous solutions of glucose but are photoreduced when incorporated in glucose glasses. All triphenylmethane dyes become increasingly more fluorescent the greater the viscosity of the medium. With those dyes which are photoreducible in glucose glass there is superposed on the room temperature fluorescence an α-phosphorescence (delayed fluorescence). In the more rigid glasses the phosphorescence lifetime decreases with increasing temperature but is almost independent of viscosity. The rate of photoreduction of C.I. Acid Violet 19 in glucose as a function of temperature is maximal at about 60°c. The kinetic and spectral data suggest that three processes must be considered: (1) suppression of internal conversion of the single excited state to the ground state by increasing the viscosity of the medium, (2) transitions from the metastable to the ground state, and (3) reaction between dye molecules in the metastable state and glucose. The first process is formed by lowering the temperature, the reverse being true for the second and third processes. C.O.C.

Photoreduction of Acridine Dyes
F. Millich and G. Ostrer
J. Amer. Chem. Soc., **81**, 1357–1363 (20 March 1959)

Some acridine dyes when irradiated by blue light in presence of allylthiourea are reduced to the leuco dyes, those having amino substituents in both the 3- and 6-positions being rapidly reduced. Another group of acridine dyes is only reduced at one tenth the rate of the first while a third group are not reduced. Relationship exists between phosphorescence of the dyes and their ability to be photoreduced. The detailed kinetics of photoreduction of proflavin at its pH for maximum rate, i.e. pH 4, coupled with fluorescence studies showed that (1) reduction proceeds through a long-lived excited state, (2) transition from the first singlet excited state to the long-lived state is induced by dye molecules in the ground state, and (3) the inductive forces of interaction act over distances of 500 A. C.O.C.

Fluorescence of Some Coumarins
C. E. Wheelock
J. Amer. Chem. Soc., **81**, 1348–1352 (20 March 1959)

The ultraviolet absorption and fluorescence intensity at various concentrations in alcohol, fluorescence spectra and stability of fluorescent intensity in alkaline solution have been determined and studied for the effect of constitution on the fluorescence of coumarins. The effect of substituents on fluorescence intensity and on position of the fluorescence band maximum was also noted. Concentration quenching and ultraviolet absorption are related. Structures hindering cleavage of the heterocyclic ring by alkaline solution stabilise fluorescence. C.O.C.

Synthesis of Cyanine Dyes by the Condensation of Diethylaminobenzaldehyde with Appropriate Heterocyclic Compounds—VIII
M. Q. Doja and J. C. Banerji
J. Indian Chem. Soc., **35**, 779–783 (Nov. 1958)

Diethylaminobenzaldehyde has been condensed with the methiodides of 2,4-, 2,5- and 2,7-dimethylbenzothiazole and with 2-methyl-6-iodo-benzothiazole to give reddish blue cyanine dyes. The order of extra sensitisation is 4- > 6- > 7-methyl > unsubst. > 5-methyl. The 4,6-dimethyl derivative is a weaker sensitiser than either the 4-methyl or the 6-methyl compound. C.H.R.

2-Aminobenzothiazoles
P. N. Bhargava and B. T. Baliga
J. Indian Chem. Soc., **35**, 807–810 (Nov. 1958)

2-Aminobenzothiazole and its halogeno, alkyl, alkoxy, and nitro derivatives and 2-aminonaphthothiazoles have

been prepared and oxidised to their azo compounds with sodium hypochlorite solution. C.H.R.

Synthesis of 8-Phenyl*meso*benzanthrone
N. Campbell, R. F. Neale, and R. A. Wall
J.C.S., 1409–1412 (April 1959)

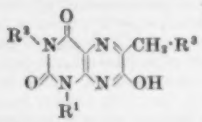
Two methods are described for the synthesis of 8-phenyl*meso*benzanthrone which do not involve the use of aluminium chloride viz., by the condensation of 1-oxoperinaphthene-2-carboxylic acid with 1-phenylbuta-1,3-diene, and by heating 1-phenylphenanthraquinone (I) with sulphuric acid, ferrous sulphate, and glycerol; if 1-phenylanthraquinone is used instead of I, the reaction is unsuccessful owing to sulphonation of the phenyl nucleus. H.H.H.

Perpendicular Conjugation in Some Octahedral Metallophthalocyanine Derivatives
J. A. Elvidge and A. B. P. Lever
Proc. Chem. Soc., 123–124 (April 1959)

Manganous phthalocyanine reacts with methanolic KCN to yield hydroxycyanomanganese phthalocyanine and with methanolic NaOH to afford dihydroxymanganese phthalocyanine (I), while chromium phthalocyanine (obtained from chromic acetate and phthalonitrile) gives aquohydroxychromic phthalocyanine (II) with aq. ethanolic acid, and this cpd. reacts with methanolic NaOH to give disodium oxyhydroxychromium^{III} phthalocyanine. These cpd. are true octahedral (6-co-ordinate) metal complexes and are the first examples to be prepared in the phthalocyanine series; I and II are unique in being dibasic acids. The considerable stabilisation of the anions in the above cpd. is ascribed to perpendicular conjugation between the mutually perpendicular phthalocyanine and oxy-groups, which appears possible through p_π–d_π orbital overlap and is supported by ultraviolet and visible absorption data. H.H.H.

Pteridines from 4,5-Diaminopyrimidines and α-Thioketo Acids—II
S. N. Baranov and N. E. Tarnavskaya
Ukrain. khim. zhur., **24**, (4), 472–476 (1958)

Condensation of equimolar amounts of diaminopyrimidines with α-thioketo acids in 96% and 50% ethanol has yielded 14 pteridines of general formula—



(R^1 = phenyl, *p*-tolyl, or *o*- or *p*-anisyl; R^2 = H or CH_3; R^3 = C_6H_5 or 4,3-$C_6H_3(OH)(O{\cdot}CH_3)$. The fluorescent colour and other physical data are given, and the absorption spectra (220–360 mμ.) for 8 pteridines are shown. G.J.K.

PATENTS

Anthraquinone
Koppers Co. *USP* 2,865,933

Anthraquinone is made in high yield and of high purity by oxidising molten anthracene or anthracene dissolved in an inert solvent, e.g. nitrobenzene, with 98% HNO_3 at temperatures > 150°c. Anthracenes of 84·7% and 95·8% purity yield anthraquinones of 98·3 to 99·2% purity without further purification. E.T.

4-Arylamino-2-Hydroxyquinolines
Gy *BP* 811,957

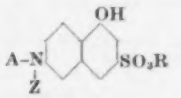
Dianilides of malonic acid in which the anilide groups may carry Alk or Hal substituents (except in the *o*-position) are heated with $AlCl_3$ at 270–320°c. to give the title products, which may be converted into substituted 2,4-dihydroxyquinolines for use as coupling components for azo dyes. Thus the bis-*m*-chloroanilide of malonic acid gives 4-*m*-chloroanilino-7-chloro-2-hydroxyquinoline, converted by heating at 200°c. with 7% aq. HCl into 7-chloro-2,4-dihydroxyquinoline. E.S.

Continuous Diazotisation Process
FH *BP* 812,368

A continuous diazotisation process, applicable in general to all diazotisable amines, is described, whereby an amine or amine salt fed through a system of pumps and rotameters is mixed with a solution of a diazotising agent (e.g. sodium nitrite) fed through a similar system at controlled rates. The reaction is controlled by redox potential measurements. A diagram is given and a plant is described which holds 2 l. of liquid in the cycle and diazotises 874 g. of 32·88% aniline solution per hour. E.S.

Azine Cyan Colour Formers
General Aniline *USP* 2,871,120

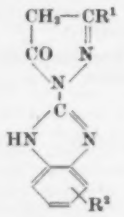
Colour formers of formula—



(A = subst. or unsubst. benzyl; Z = subst. or unsubst. benzyl or an aromatic radical of the benzene, naphthalene, pyridine or quinoline series), e.g. 6-[(*N*-*p*-nitrobenzyl, *N*-benzyl)amino]-1-naphthol-3-sulphonic acid, are more brilliant and have greater green transmission than those previously used. C.O.C.

Colour Formers producing Magenta Azomethin Dyes
Ilford *BP* 813,866

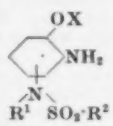
Compounds of formula—



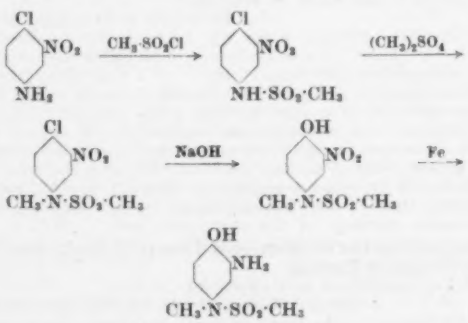
(R^1 = Alk, aralkyl, Ar or heterocyclic; R^2 = H or a substituent such as Hal, Alk or alkoxy) e.g. 2-hydrazinobenziminazole, are colour couplers which yield magenta azomethin dyes of reduced absorption for blue light. C.O.C.

Metal(Chromium and Cobalt)-complex Monoazo Dyes for Wool, etc.
FBy *BP* 813,186

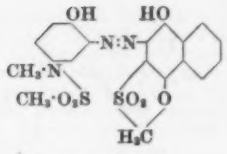
Derivatives of *o*-aminophenol



(X = H or Alk; R^1 = Alk or hydroxyalkyl; R^2 = Alk or phenyl) are diazotised and coupled with *o*-coupling hydroxy compounds free of SO_3H or COOH groups to give monoazo compounds whose 1 : 2 metal complexes with Co and Cr dye wool, nylon, etc. from neutral or weakly acid baths. A route to these diazo components is e.g.



This amine, diazotised and coupled with 1:4-naphthohydroquinone methylene sulphone ether gives



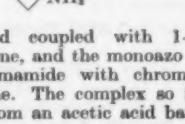
whose Cr-complex dyes wool blue. E.S.

Metal(Chromium and Cobalt)-complex Monoazo Dyes for Wool, etc.

Gy *BP* 812,151

Monoazo dyes X^1·A·N:N·B·X^2 (A = benzene residue; B = naphthalene or pyrazolone residue; one X = metallisable group; the other X = $-(SO_2)_n$·NH·D; D = an organic radical containing a group taking part in the metal-complex formation; n = 0 or 1) are treated with "co-ordinative divalent complex formers" in the presence of Co and Cr compounds to give water-soluble complexes which dye wool, nylon, etc. from neutral or weakly acid baths. The co-ordinative divalent complex formers include o-hydroxycarboxylic acids, 8-hydroxyquinolines, o-hydroxyazo compounds, etc., and their presence in the dye influences levelling by "masking" the metal present and so preventing too rapid fixation of dye to fibre.

The monoazo compounds are prepared (1) by coupling diazotised derivatives of orthanilamide, which contain a propionic acid, or o-hydroxy(or carboxy)phenyl radical as substituent on the amide N atom, with 5-pyrazolones or o-coupling naphthols, and (2) by coupling diazotised o-aminophenols or anthranilic acid with o-coupling amines whose N atom carries an o-hydroxy(or carboxy)phenyl radical, e.g. 2-(o-hydroxyphenylamino)naphthalene. Thus the o-carboxyanilide of orthanilic acid

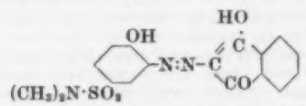


is diazotised and coupled with 1-m-chlorophenyl-3-methyl-5-pyrazolone, and the monoazo compound heated at 135°C. in formamide with chromium acetate and 8-hydroxyquinoline. The complex so formed dyes wool greenish-yellow from an acetic acid bath. E.S.

Metal(Chromium and Cobalt)-complex Monoazo Wool Dyes from Indandione(1,3)

FBy *USP* 2,868,777

Diazotised o-aminophenols having a sulphamyl or alkylsulphonyl substituent are coupled with indandione (1,3), or with derivatives such as its 2-carboxylic acid which form indandione(1,3) under the conditions of coupling, to give monoazo compounds whose Co and Cr complexes dye wool, nylon, etc. brown from neutral or weakly acid baths. Thus diazotised 2-aminophenol-4-sulphondimethylamide is coupled with aq. suspension of indandione(1,3), adding aq. ammonia dropwise until reaction is complete, to give

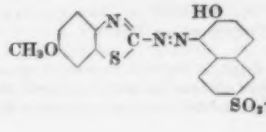


Dissolving in aq. NaOH and stirring at 90–95°C. with aq. $CoCl_2$ gives the complex, which dyes wool red-brown from a neutral bath. E.S.

Metallisable Monoazo Disperse Dyes

Eastman Kodak Co. *USP* 2,868,774

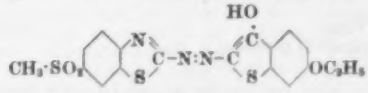
Diazotised 2-aminobenzthiazoles are coupled with 2-naphthol-6-sulphonamides to give disperse dyes which may be aftertreated on the fibre with metal salts (especially $Ni(SCN)_2$) to improve their fastness. Thus diazotised 2-amino-6-methoxybenzthiazole is coupled with 2-naphthol-6-sulphondi(β-hydroxyethyl)amide to give



which dyes cellulose acetate reddish-orange, converted to bright blue by padding with 2% aq. $Ni(SCN)_2$, drying, and steaming 10 min. at 5 lb. p.s.i.

USP 2,868,775

Similar dyes are made by coupling diazotised 2-amino-benzthiazoles with 3-hydroxythianaphthenes. Thus diazotised 2 - amino - 6 - methylsulphonylbenzthiazole coupled with 6-ethoxy-3-hydroxythianaphthene gives



which dyes cellulose acetate yellow, becoming bright red-violet when aftertreated with $Ni(SCN)_2$. E.S.

Brown, Copperable Disazo Direct Dyes

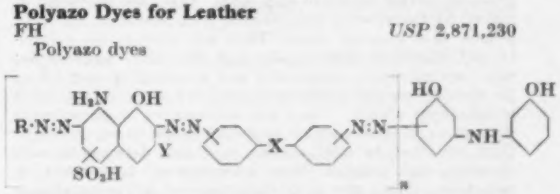
FBy *BP* 812,102

Tetrazotised 3-hydroxy(or alkoxy)benzidine, which may carry a 3′-alkyl group, is coupled with 1 mol. of salicylic acid or a derivative thereof and then with 1 mol. of an N-aryl-γ acid which contains a water-solubilising group in the aryl radical, to give brown direct dyes which may be coppered in substance or on the fibre. Thus tetrazotised 3-ethoxybenzidine is coupled first with 1 mol. of salicylic acid in presence of Na_2CO_3 and then with 1 mol. of N-m-sulphophenyl-γ acid in presence of pyridine. Heating at 120–160°C. with aq. $CuSO_4$ gives the complex, which dyes cotton dark red-brown. E.S.

Polyazo Dyes for Leather

FH *USP* 2,871,230

Polyazo dyes



(R = Ar of benzene or naphthalene series; X = direct link, CH:CH, NH·CO·NH, CO, O, S, NH, or CONH; Y = H or SO_3H; n = 1 or 2; at least two SO_3H groups are present) give violet, dark blue, dark green or black on leather. An example is

sulphanilic acid $\xrightarrow{1}$ H acid $\xleftarrow[alkaline]{2}$ benzidine $\xrightarrow{3}$ 4,4′-dihydroxydiphenylamine

which dyes leather deep bluish-black. E.S.

Anthraquinone Azo Pigments

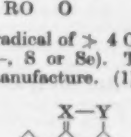
General Aniline *USP* 2,871,234

Oxidising, preferably with $NaNO_2$, leuco sulphuric esters of anthraquinone azo compounds obtained by coupling diazotised leuco sulphuric esters of amino-anthraquinones with pyrazolones, acylacetarylides, or arylides of o-hydroxycarboxylic acids, in presence of a poly-N-vinyl-α-pyrrolidone gives pigments of improved brightness. E.S.

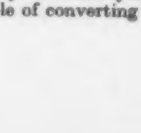
Anthrone Disperse Yellows

Ciba *BP* 812,825

Yellows of high light fastness on cellulose esters, polyamides, polyurethanes or Terylene are obtained using dispersed anthrones of formula—



(R = H, aliph. acyl radical of > 4 C; X = N or –CH=; Y = –NH–, –N Alk–, S or Se). There are two routes available for their manufacture. (1) A compound



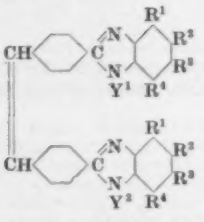
(Z = NH_2, OCH_3, oxy or selenocyano group) is treated with a reagent capable of converting the group Z to OH.

For example, when Z is an amino group, by treatment with aq. HNO_2 at 20–30°c. followed by boiling. (2) A 5-hydroxyanthraquinone having, in the 1-position, a Hal, SO_2H, SO_2Cl, NH_2, $NHNH_2$, CNS or CNSe group is reacted with, e.g. Na *poly*sulphide and ammonia, to give a 5-membered heterocyclic ring fused on in the 1,9-position. The 5-hydroxy group may be acylated. E.T.

Benzimidazolylstilbene Fluorescent Brightening Agents

Sterling Drug *BP* 813,543

Compounds of formula—

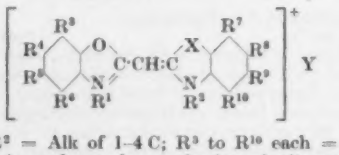


(R^1, R^2, R^3 and R^4 = same or different H, Alk of 1–4 C, alkoxy, or Hal; Y^1 and Y^2 = same or different H, Alk of 1–6 C, hydroxyalkyl of 2–6 C, hydroxy-oxaalkyl of 3–15 C, carboxyalkyl of 2–6 C, cyanoalkyl of 3–6 C, or subst. or unsubst. monocyclic aralkyl of 7–11 C) are yellow to greenish yellow solids of high m.p. They are insoluble in water, hydrocarbons, halogenated hydrocarbons, ketones, ethers, and mineral acids. They are moderately soluble in *NN*-dimethyl formamide and dimethyl sulphoxide; some are soluble in acetic acid and where Y^1 and/or Y^2 = H, they are weakly acidic and yield yellow to red solutions in alcoholic alkali. They are substantive from aqueous dispersion to most natural and man-made fibres on which they are fast to light, soap, synthetic detergents, and chlorine and exhibit blue fluorescence in ultraviolet radiation. Thus the acid phthalate of 4-(benzimidazol-2-yl)-4′-[1-(2-hydroxy-2-sulphopropyl)benzimidazol-2-yl] stilbene is a useful fluorescent brightening agent for application to textiles from soap or other detergents. 38 other examples are given. C.O.C.

Fluorescent Brightening Agents for Acrylonitrile Polymers

Gy *BP* 813,843

Compounds of formula—

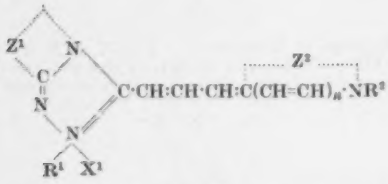


(R^1 and R^2 = Alk of 1–4 C; R^3 to R^{10} each = H or a nonionogenic and nonchromophoric substituent; X = S or a hydrocarbon radical containing the bonds shown at the same C atom; Y = anion) have good affinity for acrylonitrile polymers and copolymers on which they exhibit greenish to violet blue fluorescence. Thus (3-methylbenzthiazolo)-(3-methylbenzoxazolo)-monomethin cyanine-*p*-toluene sulphonate exhausts from aqueous solution on to Orlon type 42 at the boil. C.O.C.

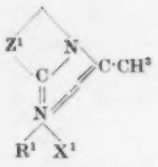
Cyanine Dyes from Triazolo Bases

Eastman Kodak Co. *USP* 2,870,014

Dyes of formula—



(R^1 and R^2 = Alk; n = 0 or 1; X^1 = acid radical; Z^1 = atoms to complete a benzothiazole or quinoline nucleus; Z^2 = atoms to complete a 5- or 6-membered nucleus) are obtained by condensing a compound of formula—



with one of formula—

(R^3 = carboxylic acyl group; R^4 = Ar). Thus 1-methyl-s-triazolo[3,4-b]benzothiazole was heated with methyl *p*-toluenesulphonate at 160–170°c. for 3 hr. It was then cooled and 2-β-acetanilidovinylbenzoxazole ethiodide added together with pyridine and triethyl amine and the whole refluxed for 10 min. This yielded the scarlet dye, 3-ethyl-2′-methyloxa-1′-s-triazolo[3,4-b]benzothiazolo-carbocyanine iodide. C.O.C.

β-Carotene Derivatives— Food and Cosmetic Dyes

California Institute Research Foundation *USP* 2,871,267

β-Carotene derivatives having varying spectral characteristics and useful for colouring food and cosmetics are produced by treating β-carotene (or other β-ionylidene polyene) with a *N*-bromo compound, e.g. *N*-bromo-succinimide, in a chloroform–alcohol mixture to form bromo and ketal derivatives which are then treated with an organic base and the resulting derivatives separated by chromatography. Thus β-carotene treated in the above manner yields mainly 4-diethylketal-4′-bromo-β-carotene but 4-diethylketal-β-carotene, 4-bromo-β-carotene, 4,4′-tetraethylketal-β-carotene and 4,4′-dibromo-β-carotene are also formed. On treatment with excess *N*-phenyl-morpholine a mixture of the following is produced. 4-keto-3′,4′-dehydro-β-carotene, 4-keto-β-carotene, 4,4′-diketo-β-carotene, 3,4,3′,4′-bisdehydro-β-carotene, and retro-bisdehydrocarotene. C.O.C.

Modified Cadmium Sulphide Pigments

Harshaw Chemical Co. *BP* 813,952

Red pigments having quality, tonal, and pigment characteristics similar to those of cadmium sulphoselenide pigments are obtained by calcining a mixture of mercury sulphide, cadmium sulphide, and an alkali metal sulphide. The latter acts as a catalyst promoting formation of a solid solution of Cd and Hg sulphides, the product obtained by heating these two sulphides together in absence of this catalyst being a very inferior product. C.O.C.

Titanium Dioxide for Pigmenting Fibres and Films of Synthetic Polymers

National Lead Co. *USP* 2,868,663

Rutile TiO_2 coated first with 0·25–10·0% of a hydrous oxide of Al, Ti, Si or Zr and then with 0·2–5·0% of a compound of formula—

$$R^1{\cdot}CHOH{\cdot}COOR^2$$

(R^1 = Alk or Ar, R^2 = H, NH_4, alkali metal, Alk or Ar), e.g. lactic acid, is readily dispersed in organic solvents. When added to organic solvent solutions of macromolecular polymers it is uniformly dispersed and fibres or films produced from the pigmented solution have consistent and uniform colour which does not vary from batch to batch. C.O.C.

Densification of Carbon Blacks (I p. 431)
Microdispersions of Photographic Colour Couplers (IX p. 439)
Paper-coating Pigments (XI p. 441)
Autoxidation Reactions in the Presence of Light-sensitised Chlorophyll (XIII p. 442)
Relationship between the Affinity of Direct Dyes and their R_f Values (XIV p. 444)

V—PAINTS; ENAMELS; INKS

PATENTS

Aqueous Stencil Duplicating Ink

A. B. Dick Co. *USP* 2,868,741

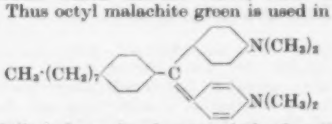
A rapid drying duplicating ink contains a colorant, water as the principal diluent, a water-soluble high mol. wt. compound to impart body, and an aqueous dispersion

of a water-insoluble organic material to protect the copy after it is dried, e.g. 16% (by wt.) Carbon Black (C.I. Pigment Black 6 and 7), in 40% aqueous dispersion, 2% dioctyl ester of sodium sulphosuccinate in 50% solution, 40% polyvinyl chloride in 55% aqueous dispersion, and 42% hydroxyethyl cellulose (high-viscosity grade) in 10% aqueous solution (400–500 cps. in 5% solution at 20°c.). C.O.C.

Wood Stains and Lacquers containing Alkylated Basic Dyes

Midland Chemical Corpn. *BP* 813,921

Basic dyes containing an alkyl radical of 8–12 C not only stain wood but also render it water-repellent. Dissolved in ether alcohols they readily penetrate wood to give stains which neither bleed, fade nor raise the grain. Thus octyl malachite green is used in a



mixture of diethylene glycol monoethyl ether (1 part by vol.), methyl alcohol (9), and toluol (6). These dyes can also be used for colouring lacquers, particularly nitrodextran lacquers used for nail varnish. C.O.C.

Pigmented Lacquers containing Cellulose Nitrate and Oil-modified Alkyd Resin

DuP *BP* 813,892

A lacquer of improved outdoor durability comprises pigment, volatile organic solvent, and organic film-forming material consisting essentially of (1) a butyl methacrylate-alkyd resin copolymer and (2) lacquer-grade cellulose nitrate the proportions of these two ingredients being 1 : 1 to 3 : 1. The copolymer contains in each 100 parts by weight (*a*) 20–60 parts of butyl methacrylate and (*b*) 80–40 parts of a phthalic glyceride alkyd resin of acid number < 10 and containing chemically combined with it 40–55% of coconut oil, 1–8% of maleic anhydride and unesterified hydroxyl groups equivalent to 0·4–10·2% of glycerin. C.O.C.

Silica Flatting Agents

Monsanto Chemical Co. *USP* 2,870,035

An amorphous, hydrophilic yet water-insoluble silica material of acid number $> 0{\cdot}2$ and containing > 1000 silanol groups is treated with an organic silicate containing at least two –OR groups (R = Alk, Ar, aralkyl, or alkaryl) attached to a Si atom which is also attached to at least one other O atom, until a hydrophilic to partly hydrophobic but non-organophilic product is produced. The product is readily incorporated into non-aqueous coating compositions, particularly cellulose nitrate lacquers, which yield clear, transparent flatted or semi-gloss coatings having a smooth surface. C.O.C.

Aqueous Coating Compositions for Use on Non-porous, Rigid Substrates

Rohm & Haas Co. *BP* 813,947

Hard non-blocking coatings are obtained by use of aqueous dispersions of polymers having T_1 value $\not< 35$°c. Thus an aqueous dispersion containing a 10 : 90 (by wt.) ethylacrylate-methylmethacrylate copolymer of T_1 80°c. (50 parts per 800 parts of dispersion) and the half amide half-ammonium salt of a 1 : 1 molar copolymer of maleic anhydride with di-*iso*butylene is coated on to asphalt tiles and dried. This yields a hard abrasion-resistant coating which shows no blocking at 170°F. and 75% R.H. when the coated tiles are stacked on one another under a pressure of 10 lb./sq.in. C.O.C.

Paper-coating Pigments (XI p. 441)
Effect of Permanent Red F5R on Electrical Insulation Resistance (XIII p. 442)

VI— FIBRES; YARNS; FABRICS

West German and English Textile Research 1957

H. Zahn and G. Satlow

Melliand Textilber., 40, 471–480 (May 1959)

Comparisons of published work lead the authors to conclude that fundamental work concerning fibres is more emphasised in England, whereas subsequent processing receives more attention in Germany. Great Britain, they feel, has better facilities as regards professorial chairs, number of scientists, lengthy projects, and expensive instruments. Over seven columns of useful classified references for the year are appended. S.M.J.

Structure of α-Keratin

R. D. B. Fraser, T. P. Macrae, and G. E. Rogers

Nature, 183, 592–594 (28 Feb. 1959)

Electron-microscope observations are combined with parallel studies of low-angle X-ray diffraction and infrared dichroism. Microfibrils and matrix are observed in keratin fibres. Areas of near hexagonal packing are interspersed with regions of alternating light and dark bands. Possible interpretations are suggested. X-Ray studies suggest that the microfibrils are crystalline and the matrix largely non-crystalline. Treatment with osmium tetroxide suggests that the (A + B) fractions of the cystine residues is concentrated in the matrix. Important differences between crystallinity in cellulose and in keratin are pointed out. Rough estimates of the relative proportion of microfibrils and matrix are made. The analogy between globular protein crystals and α-keratin is emphasised. The process of keratinisation inhibits crystallisation of the fibrils. It is suggested that a transition occurs from spiral aggregates to more stable hexagonal or monoclinic forms as the size of the aggregate increases. W.R.M.

Bulked Yarn Trade Index

Silk and Rayon, 33, 137–149 (Feb. 1959)

PATENTS

Improving the Dimensional Stability of Polypropylene Fibres

Montecatini Societa Generale per l'Industria Mineraria e Chimica *BP* 813,891

Fibres of crystallisable polypropylene are oriented by stretch drawing and are then heated while kept under slight tension, the temperature and time of heating being such that if they were not under tension they would shrink considerably. C.O.C.

Fibre-forming Polythiolesters by Interfacial Condensation Polymerisation

DuP *USP* 2,870,126

Organic dithiols react with dicarboxylic acid halides smoothly and rapidly to form fibre-forming polythiolesters at moderate temperatures if they are brought together in presence of an acid acceptor in such a way that the reaction zone is at, or immediately next to, a liquid–liquid interface. Most of the molecules of at least one of the reactants must diffuse through liquid diluent to reach the reaction zone. C.O.C.

Titanium Dioxide for Pigmenting Fibres and Films of Synthetic Polymers (IV p. 436)
Shrinking and Setting Acrylonitrile Polymer Fibres and Yarns (X p. 439)
Separation and Analysis of Scale-rich Material from Wool (XIV p. 444).

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Oiling, Milling, and Scouring Wool Goods

P. Graf *Melliand Textilber.*, 40, 290–294 (March), 409–412 (April 1959)

A very thorough review (12 references) with some quantitative results. The importance of the internal pH of the wool in wet processing is emphasised, with particular reference to hydration and swelling. With soap, "soda shock" treatment is advocated for maximum scouring efficiency. The author then discusses scourability, emulsification, corrosive effect, and flammability of oleins, and pays special attention to scourability of mineral oils. Diverging desiderata for this and lubrication in spinning are noted. Special difficulties, such as reduced scourability are encountered, with afterchromed goods, owing to the formation of insoluble chromium lakes with anion-active emulsifiers. Experimental results illustrate the dependence of residual oil content on the conditions of dyeing, milling, and scouring. Discrepancies between theories of milling and findings in practice are underlined.

Examples from practice include softer handle and lower strength loss for rapidly milling olein-containing goods obtained with the more slowly saponifying triethanolamine instead of soda, and non-ionic milling assistants for chrome-tow-dyed mineral-oil-lubricated goods, as soap produced low rubbing fastness. The effect of prescouring on waterproofing with paraffin wax and Al salts is illustrated. Non-ionics are worst, and an olein lubricant best, with soap or alkylarylsulphonates as scouring and milling agents. S.M.J.

Dry Cleaning—I
R. Mönch *Faserforsch. und Textiltech.*, **10**, 129–137 (March 1959)

Technical processes of dry cleaning are briefly surveyed. The structure of non-aqueous solutions of detergents is considered and compared with that of aqueous solutions. Take-up of water by non-aqueous solutions is studied and estimated in terms of densities and viscosities of solutions. A mechanism of water absorption is developed for use in further studies. W.R.M.

PATENTS

Preventing Precipitation of Insoluble Metal Compounds in Textile Treatment Baths
BASF *BP* 813,929

Efficient prevention of precipitation both during treatment and rinsing is obtained if the bath contains both enough sequestering agent to render all hardness-causing ions present innocuous and a dispersing agent for insoluble compounds formed by hardness-causing ions but which does not itself form an insoluble metal compound. The amount of this dispersing agent is such that on rinsing with water containing hardness-causing ions the resultant metal compounds are kept in dispersion. C.O.C.

Continuous Scouring and Bleaching of Cotton Cloth
DuP *USP* 2,868,615

The wet out and desized cloth is passed either as a rope or in open width through an aqueous bath containing by weight 1·25–2·0% H_2O_2, 1·5–3·0% Na silicate of $Na_2O : SiO_2 :: 1 : 2·4$, 0·3–0·8% NaOH, a water soluble boron salt equivalent to 0·25–2·0% borax, squeezed to a moisture content of 75–150% of its dry weight, heated in presence of steam to 200–212°F. and then kept at 180–212°F. for 45–90 min. This yields a satisfactory bleach with only one continuous storage stage. C.O.C.

VIII— DYEING

Azo Dyes derived from Benzo-1,4-dioxan
H. C. A. van Beek, P. M. Heertjes, and B. J. Knape *Melliand Textilber.*, **40**, 417–419 (April 1959)

Thirty-seven azo acid dyes derived from benzo-1,4-dioxan were applied to wool and nylon. Seven of the dyeings were also afterchromed. Colour, fastness to light, washing, and perspiration, and levelling properties are tabulated. Some are claimed to be eminently suitable for nylon, giving bright, level dyeings of high fastness. There are 8 references. S.M.J.

PATENTS

Azoic Dyeing
FH *BP* 813,986

The goods are treated with an alkali metal compound of an azoic coupling component and then with an azoic diazo component, a chromic salt of a weak organic acid and an alkali metal phosphate in a bath which at the start of coupling is at pH 5–7. This gives complete coupling with no loss of the diazo component. Thus cotton yarn is treated for 30 min. at 30°C. and liquor ratio 20 : 1 in a bath containing 1-(3′-hydroxydiphenylene oxide-2′-carboylamino)-2,5-dimethoxybenzene (1·5 g. per litre), 38°Bé. NaOH (16 c.c.) and oleic acid chloride–protein-degradation condensate obtained according to *BP* 413,016 (3 g.). It is then squeezed and developed for 25 min. and liquor ratio 20 : 1 in a solution at pH 6·0–6·8 containing the diazonium chloride zinc chloride double salt of 1-amino anthraquinone (1 g./l.), chromic acetate (5), Na hexametaphosphate (1·2) and NaH_2PO_4 (0·9). After rinsing and the usual aftertreatment a deep reddish brown dyeing is obtained. C.O.C.

Colouring Cellulose Triacetate
BrC *BP* 813,610

When applying dyes, especially disperse dyes, from aqueous baths to cellulose acetate of acetyl value $< 59\%$ (calc. as acetic acid) dyeing is quicker and improved fastness to washing is obtained if the dyeing is done at $> 100°C$. C.O.C.

Formaldehyde as Assistant in the High Temperature Dyeing of Polyester Fibres
DuP *USP* 2,869,969

Dyeing with disperse dyes in a bath containing 1–10 g./l. formaldehyde at pH 4·0–5·5 at 250°F. yields greater colour value, brighter dyeings and less staining of any protein fibres present. Blends of polyester fibres with wool may have the wool dyed with acid dyes before or after the polyester fibres are dyed at 250°F., the formaldehyde protecting wool against damage at that temperature. C.O.C.

Dyeing Dacron and Dynel
Ciba *USP* 2,872,279

A mixture of *o*-phenylphenol, pine oil, and Turkey Red oil (about 50% sulphonated) in the proportions of 10 : 8 : 7 when added to the dyebath disperses in it at once without any precipitation or separating out. Added to the bath when dyeing Dacron staple fibre, either as loose fibres or tops, with disperse dyes it results in better dyeings and leaves the fibres with a soft and lofty handle. C.O.C.

Printing or Colouring Glass Fibre Fabric (IX below)
Relationship between the Affinity of Direct Dyes and their R_f Values (XIV p. 444)

IX— PRINTING

Storage of Backgreys for Roller Printing
R. Kauschka *Melliand Textilber.*, **40**, 419–421 (April 1959)

A device for storing backgreys in open width behind roller printing machines is described and illustrated. Fabric is introduced into the bottom of the pile through a slit from below, and is supported on two cloths which are moved on rollers by a pendulum. Piling is precise and compact— up to 800 metres in a pile— and delivery from the top of the pile obviates manhandling. S.M.J.

PATENTS

Printing of Cellulose Acetate or Synthetic Polymer Fibres
Société Rhodiacéta *BP* 813,887

When printing with "plastosoluble" dyes, i.e. dyes which dye cellulose acetate and synthetic polymers by solid dissolution of the dye in the fibre without aid of any oxidation or reduction step, presence of 0·5–2·0% of an oxidising agent in the paste results in better colour value, brighter colours and improved fastness. Thus Terylene fabric printed with a paste containing Eastone Red NGLF (TE) (20 g.), diethylene glycol (100), 5% aq. $NaClO_3$ (20), water (560), and industrial crystal gum (300), dried for 2 hr. at 25°C., steamed for 15 min. at 130°C. and washed, yields intense dark red prints whereas use of a similar paste containing no chlorate yields dull orange prints. C.O.C.

Printing on Cellulose Triacetate
BrC *BP* 813,611

Triacetin and other esters of di- or polyhydric alcohols with fatty acids of < 5C are very useful for assisting the fixation of disperse dyes on cellulose triacetate. Thus satisfactory prints can be obtained from a wide range of disperse dyes by printing them on to cellulose triacetate fabric as thickened aqueous pastes containing 15–25% by weight of triacetin and then steaming at atmospheric pressure. C.O.C.

Printing or Colouring Glass Fibre Fabric
Owens-Corning Fiberglas Corpn. *USP* 2,868,668

Permanent prints or coloration are obtained if the fibres are desized, treated with a composition containing an acrylic resin (or its precondensate) and a colorant and then, after setting the composition, given a final treatment with a high receptive and adherent water-repellent. Thus glass fibre fabric desized by heat cleaning for 3 min. at 1050°F. is impregnated with a composition containing by weight 10% of ethyl acrylate polymer in aqueous dispersion, 4% Malachite Green (C.I. Basic Green 4),

0·2% rosin soap, and 85·8% water. It is then dried for 10 min. at 250°F., treated with a 1% solution of stearatochromyl chloride and dried at 250°F. The treated fabric has markedly improved strength, resistance to abrasion, and fastness to light, these properties being unimpaired by repeated washing or dry cleaning. C.O.C.

Microdispersions of Photographic Colour Couplers
Eastman Kodak Co. *USP* 2,870,012

The free acid of a coupler containing an acid group is dissolved in a water-miscible solvent. Water and a wetting or dispersing agent is then added to produce a suspension of the coupler in the aqueous medium after which the solvent is removed and the coupler dispersion added to a silver halide emulsion. C.O.C.

Reduction of Dye Absorption in Hardened Gelatin Relief Matrices
Technicolor Corpn. *USP* 2,870,704

Top Coating for a Transfer Sheet
Ditto *BP* 814,002

A mixture of paraffin or other wax and 1–25% by weight of a polyethylene resin of mol. wt. 1200–6000 is an excellent top coating for transfers, carbon paper, etc. It eliminates staining, marring of the surface, offsetting, bleeding of the dye or dusting of the dye composition. In addition it yields a coating having a high sheen which makes easy the task of inspecting the transfer material to see that all its surface has been given protection. C.O.C.

Plastic Sheets Incorporating Dye Images (XIII p. 443)
Measurement of the Resolving Power and Colour Rendering of Colour Films (XIV p. 444)

X—SIZING AND FINISHING

Synthetic Resins for Chlorine-resistant Wash-and-wear Finishing
H. Enders and G. Pusch
Melliand Textilber., **40**, 314–322 (March), 429–432 (April 1959)

The different factors responsible for chlorine retention and oxidative hydrolysis are discussed from theoretical and practical points of view. Seven illustrations summarise experimental comparisons of finishes based on dimethylolurea, hexamethylolmelamine, their alkoxy derivatives, dimethylolethyleneurea (DMEU), and the ethyleneurea–triazine precondensate Knittex everfit CR (Pfersee), a further eleven demonstrate the superiority of the last named product to DMEU. The effect on chlorine resistance of varying resin, catalyst, conditions of condensation, and number of scours at the boil and type of chlorine treatment are investigated by determining chlorine retention iodimetrically, browning on ironing, chlorine damage as reduction of tear strength after ironing, and loss of crease-resist effect. Chlorine damage and browning on ironing are parallel, and are not proportional to chlorine retention. The more basic urea and melamine resins retain more chlorine. The buffering capacity of the melamine derivatives reduces damage. DMEU, excellent on chlorination only, becomes unsatisfactory after repeated scouring at the boil, retaining more chlorine as >NH groups are formed by hydrolysis, and exhibiting disproportionately greater damage, as it lacks buffering capacity, although, as it is wash-fast, crease-resistance remains high. The ethyleneurea–triazine precondensate introduces latent basic buffering capacity without increasing initial chlorine retention. Ammonium salts give the worst results. Zinc and magnesium chlorides are preferred. Condensation at 140°C. for 4 min. provides chlorine resistance similar to DMEU alone condensed at 160°C. The deterioration shown on scouring by DMEU is also greatly reduced in the case of the ethyleneurea–triazine product. S.M.J.

Flameproofing Synthetic Fibres
E. Frieser *Melliand Textilber.*, **40**, 436–438 (April 1959)

A brief review with 15 references, many dealing with nylon; melting and softening points are tabulated. 28 commercial products listed refer to a previous paper concerned with cellulosics. S.M.J.

PATENTS

Sizing Synthetic Fibre Sewing Thread
American Thread Co. *USP* 2,870,045

The thread is impregnated with an emulsion which acts as a lubricant and also gives sufficient heat resistance to minimise fusion breakage during high speed sewing of heavy fabrics. A suitable emulsion is petroleum jelly (12·5%), butyl stearate (9·5), oleic acid (8·0), triethanolamine (2·5), tetrafluoroethylene resin dispersion (4·0), and water (63·5). C.O.C.

Shrinking and Setting Acrylonitrile Polymer Fibres and Yarns
DuP *USP* 2,869,974

The goods are treated with a non-aqueous solution of ethylene carbonate, propylene carbonate, and trimethylene carbonate in a diluent, e.g. a lower aliphatic glycol. Thus a batt of an acrylonitrile–methylacrylate (94 : 6) copolymer staple fibre was treated in a 23% solution of ethylene carbonate in ethylene glycol at 130°C. Immediate shrinkage occurred which converted the batt of weight 3·2 oz./sq.yd. into a felt of 11·0 oz./sq.yd.

USP 2,869,975

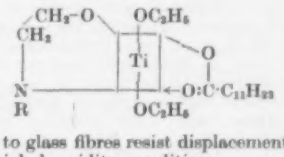
The fibres are shrunk and/or any crimp or curl imparted to them is set by treating them at < 75°C. with an aqueous solution containing 10–40% of ethylene carbonate, propylene carbonate, trimethylene carbonate, nitromethane, δ-valerolactone or γ-butyrolactone. C.O.C.

Giving Yarn an Irregular Coating of Plastic
Whiffen & Sons *BP* 813,790

Applying a plastic material containing a blowing agent which is subsequently caused to decompose and liberate gases results in an irregular coating being formed on the yarn. Suitable blowing agents are azodiisobutyronitrile, azodicarbonamide, benzenesulphonyl hydroxide, and dinitrosopentamethylenetetramine, which all decompose when heated. C.O.C.

Coating Glass Fibres
Owens-Corning Fiberglas Corpn. *BP* 813,601

Glass fibres are rendered receptive to organic materials by coating them with a complex formed by treating a titanic ester or a titanium salt of an organic acid with an alcohol, amine or hydroxyamine. Thus β-amino ethoxy titanium laurates of formula—



when applied to glass fibres resist displacement from them even under high humidity conditions.

BP 813,602

Similar results are obtained by use of a complex formed by treating an organic nitrogenous compound containing > 3 organic radicals or H atoms linked to a N atom or atoms with a hydroxy resinous polymer. Thus glass fibres are sized with a composition containing polyvinyl alcohol (2·5% by wt.), a stearic acid adduct of imidazoline (4·0), stearato chromic chloride (0·5), HCl (0·5), and water (92·5).

BP 813,603

Similar results are obtained by use of a complex formed by treating an organic nitrogenous compound containing > 3 organic radicals or H atoms linked to a N atom or atoms with polyvinyl pyrrolidone. Thus glass fibres are sized with a composition containing polyvinyl pyrrolidone (3·0% by wt.), vinylethoxy stearate (1·5), tetraethylene pentamine reacted with acetic acid (1·0), water (94·5), and acetic acid (3 c.c. per 100 g. of the composition). C.O.C.

Dimensional Stabilisation of Cellulosic Textiles
Midland Silicones *BP* 813,925

The material is impregnated with an aqueous composition containing (1) a dialdehyde, preferably glyoxal, (2) an aminoaldehyde condensate, (3) a methyl hydrogen polysiloxane, and (4) a hydroxylated methyl polysiloxane and, if desired, an acid catalyst, and is then dried and baked. This gives a better finish than use of glyoxal and an aminoaldehyde condensate alone. C.O.C.

Resin-finished Cellulosic Textiles having Improved Ironing Characteristics
Courtaulds *BP* 813,919

The fabric is treated in open width with a swelling agent which is then rinsed out. It is then dried so that it contains no creases and is then impregnated with a heat-hardening acetone-formaldehyde resin precondensate containing an alkaline catalyst and finally dried and baked. Thus viscose rayon cloth was treated for 1 min. with an aqueous solution at 20°c. containing 8% of NaOH and 8% of NaCl, rinsed with water at 80°c. and dried. It was then padded with an aqueous solution containing 0·5% of Na_2CO_3 and 20% of the precondensate obtained by heating 1 mol. of acetone, 4 mol. of HCHO, and 0·1 mol. of Na_2CO_3 for 300 min. at 25°c. Finally it was stenter dried and then baked at 140°c. for two minutes. The treated fabric when washed in soap, rinsed and drip dried was free from wet crease marks. C.O.C.

Controlling Formation of Aldehyde or Amine Odours in Fabric treated with Aldehyde Resins
West Point Manufacturing Co. *USP* 2,870,041

Treating the resin-impregnated fabric with an aqueous solution containing the HSO_3 ion prevents formation of aldehyde or amine odours. The agent supplying the HSO_3 ion may also act as the catalyst for the resin, $Na_2S_2O_5$ being particularly suitable. C.O.C.

Durable Lustre Finish
Joseph Bancroft & Sons Co. *USP* 2,870,038

Fabric impregnated with a resin precondensate is carried by a blanket into contact with the external surface of a drying drum. The pressure of the blanket against the drum is 3·5 oz. to 3 lb. per sq.in. and the drum should be at 230–300°F. The fabric is finally baked to set the resin. This imparts a subdued, highly durable lustre to the fabric. C.O.C.

Dimensional Stabilisation of Wool and other Textiles by Treatment with Volatile Polyamines and Epoxides
U.S. Secretary of Agriculture *USP* 2,869,971

Wool or other textile material is treated with a vaporised polyamine and a vaporised polyepoxide in either order. This yields a product which is virtually shrinkproof in normal laundering, while the handle, resiliency, porosity, tensile strength, and colour remain unaltered. The resistance to creasing is also improved. Thus white woollen cloth was kept exposed to the vapour of diethylene triamine for 72 hr. at 25°c. and then to vaporised butadiene dioxide for 3 hr. at 105°c. and then for 69 hr. at 25°c. The cloth picked up 8·1% by weight of diethylene triamine–butadiene dioxide resinous reaction product. It remained soft and white, and when violently agitated in an "Accelerotor" for 3 min. in 0·5% aq. sodium oleate at 40°c. in a liquor ratio of 1 : 35 showed no shrinkage. C.O.C.

Reducing the Brittleness of Regenerated Cellulose Fibres treated with a Resin Precondensate
Courtaulds *BP* 813,794

Presence of a water-soluble ketone when impregnating the fibres with the precondensate results in the treated fibres being less brittle. Thus viscose rayon staple was treated for 2 min. in an aqueous solution containing 4% of a resin precondensate prepared from 2·3 mol. melamine on 1 mol. HCHO, 1·25% softening agent, 0·75% $MgCl_2$, and 5% acetone. They were then centrifuged to remove excess solution, opened out, oven dried for 1 hr. at 100°c. and baked for 7 min. at 166°c. The water imbibition of the treated fibres was 41% and they had a good colour and were free from brittleness. C.O.C.

Flame-resistant Textiles
U.S. Secretary of Agriculture *USP* 2,870,042

Compounds containing at least two 1-aziridyl groups attached to pentavalent P react with compounds containing at least two OH groups to yield polymers containing the recurring connecting structures

$$P{\cdot}\overset{|}{N}{\cdot}CH_2{\cdot}CH_2{\cdot}\overset{|}{N}{\cdot}P \text{ and } P{\cdot}\overset{|}{N}{\cdot}CH_2{\cdot}CH_2{\cdot}OR$$

(P is pentavalent; OR = alkoxy). Applied to hydrophilic fibres these polymers reduce the combustibility and are fast to laundering. On non-hydrophilic materials they form flame-resistant coatings. Thus pentaerythritol (25 parts) was dissolved in water (200) at 30–35°c. and tri(1-aziridinyl)phosphine sulphide (25) added. Half of the solution was heated on a steam bath for 30 min. and then spread out as a thin layer and heated for 10 min. at 145°c. This yielded a light coloured product, insoluble in water and organic solvents, high flame resistant and containing S, P, and N. Cotton cloth was padded in the other half of the solution, dried in a forced draught for 10 min. at 140°c., washed and air dried. The treated cloth was very strong and flame resistant. C.O.C.

Increasing the Wear Resistance of Textiles
J. Hajdu *USP* 2,871,145

The mechanical properties of textiles are much improved by treatment first in an emulsion containing a plastic, a plasticiser and, if desired, a metal salt and then with an insolubilising agent. Thus, the first bath may be made up of equal amounts of polyvinyl chloride emulsion, paraffin wax emulsion, with a small proportion of gelatin and sodium borate, and the second bath of dilute acetic acid and a little formalin. C.O.C.

Improved Plissé Effects
United Merchants & Manufacturers *USP* 2,867,892

Superior plissé effects of improved fastness to washing are obtained if the fabric is treated with a thermosetting and/or thermoplastic synthetic resin, particularly with polyvinyl alcohol, and set to width before the caustic or other shrinkage agent is applied to produce the plissé effect. C.O.C.

XI— PAPER AND OTHER CELLULOSIC PRODUCTS

Influence of Chlorine Dioxide on the Yellowing of Cellulose
H. Sihtola and N.-E. Virkola
Paperi ja Puu, **41**, 35–41 (Feb. 1959)

Chlorine dioxide increases the carboxyl content of unoxidised cellulose, causing a decrease in D.P. and an increase in tendency to yellow. Aftertreatments, such as chlorite oxidation (I) and sodium borohydride reduction (II) further increase the carboxyl content, but I increases yellowing, while II reduces it. At pH 11·5 chlorine dioxide has no effect on cellulose oxidised with hypochlorite, but at pH 7·5 the carboxyl content increases and copper number and yellowing decrease. Subsequent hypochlorite treatment lowers the copper number and increases carboxyl content and yellowing, while borohydride reduction has little effect on carboxyl content but reduces copper number and yellowing. (*In English.*) R.A.

Influence of Substitution on the "Acidity" of Cellulose
E. Kaila and H. Sihtola *Paperi ja Puu*, **41**, 171–184 (April 1959, Special issue 4a)

Sorption experiments with ethylenediamine, pyridine, water, and acetic and formic acids showed that the influence of substituents on the "acidity" of cellulose is similar to the influence of the corresponding substituents on the acidity of low-mol.wt. compounds. The sorption of low-mol.wt. acids increased with degree of substitution (D.S.). The D.S. in derivatives from regenerated samples is slightly higher than that of the corresponding derivatives from native cellulose due to the higher accessibility of the regenerated sample. Attempts to determine "acidity" by titration in non-aq. solvents failed, only cellulose nitrate and sulphate being at all titratable. (*In English.*) R.A.

Thermodynamic Interpretation of the Swelling of Cellulose in Organic Liquids— Relation between Solubility Parameter, Swelling, and Internal Surface
E. F. Thode and G. Guide *TAPPI*, **42**, 35–39 (Jan. 1959)

A hypothesis based on Hildebrand's solubility parameter concept, has been developed to explain the swelling behaviour of cellulose in organic liquids. There is an inverse relationship between the total surface area of wood pulps dried from non-polar solvents and the solubility parameter. This agrees with the earlier thermodynamic theory if it is assumed that any non-polar solvent, dissolved in the cellulose, acts as a plasticising agent. In polar solvents there is a direct relation between per cent volume increase and the solubility parameter of the final solvent. R.A.

Application of the Kubelka-Munk Theory to the Brightness of Coated Paper
R. W. Hisey and H. W. Cobb
TAPPI, **42**, 122–124 (Feb. 1959)
The Kubelka–Munk theory of diffuse reflectance may be applied to coated papers in the normal range of brightness, provided that the coating is of reasonable weight and is well held up. If these conditions are not met, severe deviations occur. R.A.

Functions governing Coating Demand
R. M. Karapetoff Cobb

I— Introduction *TAPPI*, **41**, 581–583 (Oct. 1958)
Historical survey of investigations leading to the 1956 TAPPI Memorial Coating Conference.

II— Tests for evaluating Adhesive Demand
Ibid., 583–587
Describes tests for determining % voids in paper-coating pigments based on critical pigment volume concentration (C.P.V.C.) of the paint industry and saturation of voids in soil science, using water and kerosene as test liquids.

III— Pigment Voids. Test Results and Relative Packing Power of Water and Kerosene
Ibid., 587–590
Coating demand is entirely independent of specific surface and particle size, being primarily governed by available % voids in the pigment calculated from data on water adsorption. The min. casein requirement is met when the amount of adhesive fills the available pigment voids to such an extent that the adhesive may be described as being in the upper pendular, or lower funicular, stage in the coating matrix.

IV— Correlation of Pigment Properties with Adhesive Demand
Ibid., 590–600
Considering pigment–adhesive relationships on a volume basis, rather than weight, it was found that adhesive demand is governed by the % voids originally present in each pigment as packed by the surface tension of the water used as test medium. R.A.

Paper-coating Pigments
TAPPI Monograph Series, **20**, 1–18 (1958)
This is a completely revised edition of *TAPPI Monograph* **7**, (1948), substantially by the same authors. All the chapters include information on source, manufacture, and principal characteristics, and most of them also have some reference to the economics of the industry (145 references).

I— Introduction
W. R. Willets
Lists pigments used or suggested for paper coatings, discussing the factors affecting finish, brightness, and opacity with particular reference to pigment constituents in relation to other variables.

II— Barium Sulphate
R. E. Russell *Ibid.*, 19–26
The whiteness of $BaSO_4$ is not excelled by any other pigment, it is easier to tint and to correct, and its insolubility makes it particularly suitable for photographic use.

III— Calcium Carbonate
R. W. Hagemeyer and A. M. Brooks *Ibid.*, 27–53
Opacity and brightness of calcium carbonate coatings go through a max. in the 0·1–0·3 μ. range, but gloss does not increase as much as expected with varying particle size. Ink receptivity and adhesive demand increase with decreasing particle size.

IV— Calcium Sulphite
G. Haywood *Ibid.*, 54–56
Because of high adhesive demand and low gloss-producing value, calcium sulphite is used only in small quantities to produce bright and ink-receptive papers. Adhesive demand can be reduced and gloss-producing value increased by mechanical or chemical treatment, but this is not yet a commercial proposition.

V— Clay (Kaolin), C.I. Pigment White 19
S. C. Lyons *Ibid.*, 57–115
The extreme particle fineness of clay and its low density enable it to yield a wide range of aqueous suspensions. If clay slurries are to be pumped or freely handled in the flocculated condition, the solids content should be below 35%. If deflocculated, the slurries can be pumped and kept in agitated storage up to 71% solids content.

VI— Colorants for Aqueous Mineral Coatings
M. J. Landberge *Ibid.*, 116–129
Classification of dyes with C.I. and Prototype No. as classified by the U.S. Tariff Commission.

VII— Diatomaceous Silica, C.I. Pigment White 27
C. J. O'Neil *Ibid.*, 130–131
Because of diverse particle shapes the adhesive demand of silica to give a certain pick test is about the same as that of coating clays. The flatting effect is its outstanding property. In small quantities it eliminates gloss and gives resistance to calender darkening. Minor percentages give anti-slip characteristics, but too much may have abrasive action.

VIII— Luminescent Pigments
V. A. Belcher *Ibid.*, 131–134
Daylight-fluorescent materials, generally applied by screen methods, are mostly dye–resin combinations, the fluorescence depending on the dye being soluble in the resin at certain concn. Black-light fluorescers may be incorporated in the usual coating or varnish formulae. Phosphorescent pigments containing Ca and Sr sulphides are limited to lacquer-type coatings because of susceptibility to moisture.

IX— Satin White, C.I. Pigment White 25
E. Sutermeister and R. N. Thompson *Ibid.*, 135–156
Satin White, used for its bright, white colour, high gloss, and waterproofness in conjunction with casein, is expensive and requires much more adhesive than clay. As other, cheaper coatings are improved, its use has declined.

X— Talcs (Ultra-fine), C.I. Pigment White 26
R. S. Lamar *Ibid.*, 157–195
Newly developed grades of superior hardness and at ~5 μ. give opacity values equivalent to the best grades of coating clays with better brightness.

XI— Titanium Pigments, C.I. Pigment White 6
W. R. Willets, R. T. Bingham, and F. R. Marchetti
Ibid., 196–223
Specially useful in light-weight papers, but equally applicable to board and wallpaper, because of high opacifying and brightening effects. Relatively small amounts of titanium pigment are needed and, therefore, their effect on viscosity, adhesive ratio etc., is small.

XII— Zinc Sulphide and Zinc Oxide Pigments, C.I. Pigment Whites 4 and 7
V. A. Belcher *Ibid.*, 224–227
Especially useful in waxed papers, ZnS is compatible with the usual water-sol. adhesives. ZnO has excellent colour and smoothness and gives harder coatings, but has not as much hiding power. R.A.

Formation of Complexes during Periodate Oxidation of Polyols
T. P. Nevell; G. R. Barker
Chem. and Ind., 567 (2 May 1959)
Barker and Shaw have concluded that the rapid non-oxidative reaction of periodate at pH 7 is diagnostic for the *cis-cis*-1,2,3-triol structure, and that an intermediate complex is formed which is a triester of the triol and periodate. Nevell suggests that triester ions are formed which, by analogy with results on periodate oxidation of 1,2-diols, will not readily dehydrate and decompose, the formation of diesters (which may be formed at lower pH) being inhibited. Barker accepts the explanation, which if correct supports the view that periodate forms a triply linked complex with appropriate triols and that the behaviour described is diagnostic for the *cis-cis*-1,2,3-triol system in a six-membered ring. W.R.M.

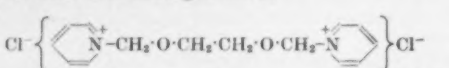
Synthesis of Cellulose in Ethanol Extracts of *Acetobacter Xylinum*
J. R. Colvin *Nature*, **183**, 1135–1136 (18 April 1959)
Ethanol extracts of *Acetobacter xylinum* contain a compound which is rapidly converted into cellulose microfibrils in water. Formation of microfibrils occurs in complete absence of bacterial cell walls and is accelerated by a thermolabile extracellular substance, presumably an enzyme, in the medium of an active culture. W.R.M.

Alkylation of Cellulose

H. Rath and V. Einsele

Melliand Textilber., **40**, 526–531 (May 1959)

The alkylation of cellulose is attempted with 23 water-soluble hydrolysable bifunctional onium compounds. Alkylation and cross-linking are diagnosed by measuring swelling, cuprammonium solubilities, and crease-resistance. The reaction is much more difficult than with other compounds containing OH, SH, or NH_2 groups, such as wool. Pyridinium and hexamethylenetetrammonium salts do not react even after 1 hr. at 160°c.; dimethylanilinium and sulphonium salts react to some extent above 120°c. If a hetero atom is present in the β-position to the central atom— as in I, from pyridine and ethylenebis-chloromethyl ether— then pyridinium salts already produce some cross-linking at 120°c.



(I)

S.M.J.

PATENT

Resin-reinforced Tissue Paper

Rohm & Haas Co. *BP* 813,548

A wet-strength tissue paper ($\leqslant$ 1·3 lb./in.) is impregnated with an aq. dispersion of an acrylic polymer or copolymer of T_1 value $\leqslant$ 45°c. to deposit 60–180% by wt. The coated paper is then dried at below the T_1 value to avoid coalescence of the polymer particles. The resulting flexible, non-blocking paper may be converted to a self-sustaining film, pellicle or glassine-type sheet by heat and pressure to coalesce the polymer. The paper is useful as self-supporting packaging material, laminating inter-layers or as overlays for modifying, decorating or protecting other surfaces. R.A.

Dry Size for Paper (III p. 432)

XII— LEATHER; FURS; OTHER PROTEIN MATERIALS

Mode of Co-ordination of Amino Acids with Cationic Chromium in Acid Aqueous Solutions— Spectrophotometric Studies

S. G. Shuttleworth and R. L. Sykes

J. Amer. Leather Chem. Assocn., **54**, 259–268 (May 1959)

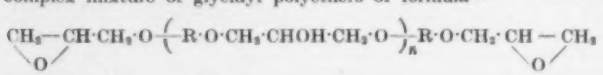
Spectrophotometric curves of Cr complexes formed with α-, β-, γ-, and ε-amino acids at 1, 2, and 3 moles/mole Cr and aged to equilibrium at pH 2·50, 3·16, and 3·82, show that the molar extinction coefficients increase from the α to the ε forms, i.e. in opposition to the effect which would be expected from chelation of both carboxyl and amino groups. The extent of co-ordination appears to be approx. proportional to the pK of the carboxyl groups, which are influenced by the number of carbon atoms separating the carboxyl and amino groups. A similar pattern is revealed by studying the fixation of Cr by pelt from soln. masked with α-, β-, and γ-amino-n-butyric acids, the masking power increasing from α to γ. The results also accord with the view that the stability of co-ordination of an acidic group to Cr is inversely related to the dissociation constant of the acid. J.W.D.

PATENTS

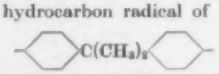
Tanning with Epoxy Resins

U.S. Secretary of Agriculture *USP* 2,869,970

The condensate of epichlorohydrin with 2,2-bis(4-hydroxyphenyl)propane, is usually not a simple but a complex mixture of glycidyl polyethers of formula—



(R = divalent hydrocarbon radical of



n = 0–10). Dissolved in a water-miscible organic solvent containing 4–35% of water it is used at pH 7–12 for tanning skins to produce leather which is white throughout its thickness. Use of inorganic salts to control the plumping action on the skin is unnecessary, alteration in the amount of water present sufficing for this purpose. The tanned leather is very resistant to the action of detanning agents C.O.C.

Impregnating Leather with Polyesters or Polyesteramides in presence of Polyfunctional Isocyanates

ICI *BP* 815,185

Leather has its resistance to abrasion much increased and its impermeability to oils and water improved by impregnating it with a polyester or polyesteramide in presence of a polyfunctional isocyanate. Curing takes place in 48 hr. at room temperature but temperatures up to 70°c. may be used to reduce the curing time. C.O.C.

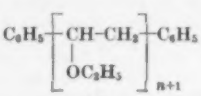
XIII— RUBBER; RESINS; PLASTICS

Polyene Compounds. III— Synthesis of Diarylpolyene Hydrocarbons

B. M. Mikhailov and L. S. Povarov

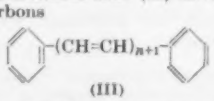
Izvestiya Akad. Nauk S.S.S.R., otdel. khim. nauk, (2), 314–319 (Feb. 1959)

The action of benzylmagnesium chloride on phenylpolyethoxyalkanes yields diphenylpolyethoxyalkanes of the form



(I)

The action of aq. alcoholic HBr (II) on I yields diphenylpolyene hydrocarbons



(III)

With phenylethoxyaldehydes the above Grignard reagent gives diphenylhydroxypolyethoxyalkanes, which with II also give III. G.J.K.

Autoxidation Reactions in the Presence of Light-sensitised Chlorophyll

N. A. Khan

Pakistan J. Scientific and Industrial Res., **1**, 258–264 (Oct. 1958)

Methyl oleate and methyl linoleate are rapidly oxidised by air in the presence of light and chlorophyll. Without either light or chlorophyll the rate of autoxidation is lower, and the induction period is very marked. Light-sensitised chlorophyll does not catalyse the oxidation of saturated hydrocarbons. E.V.T.

Effect of Permanent Red F5R on Electrical Insulation Resistance

N. Nakajima and H. Hirohata

Shikigai Kyokaisha, **30**, 54–6 (1957): *Chem. Abs.*, **53**, 4768 (10 March 1959)

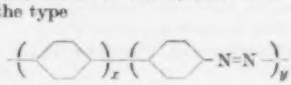
Addition of the Ca lake of Permanent Red F5R to polyvinyl chloride sheeting containing dioctyl phthalate increases the surface resistance but not the volumetric resistances. This is probably caused by adsorption of decomposition products of polyvinyl chloride by the pigment. Appreciable amounts of a 1 : 1 mixture of NaCl and $CaCl_2$ were added to the polyvinyl chloride without affecting either its surface or volumetric resistance. It seems that these salts cannot be moistened because of the protective covering of polyvinyl chloride; even if moistened and then mixed, the resulting ions cannot move within the polyvinyl chloride. C.O.C.

Synthesis of Polyphenylenes

V. P. Parini and A. A. Berlin

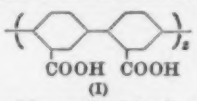
Izvestiya Akad. Nauk. S.S.S.R., otdel. khim. nauk, (12), 1499 (Dec. 1958)

The action of cuprous salts on 4,4′-benzidine-bisdiazonium salts leads to the formation of an infusible polymer of the type



Bis diazotisation of 3,3′-benzidine dicarboxylic acid, with

subsequent decomposition of the bisdiazo cpd. by the above method yields a soluble infusible polymer



(I)

giving a new method for synthesising linear polyphenylenes and their derivatives. Hard films are obtained from solutions of I. G.J.K.

Chemical Transformations of Unsaturated and High-molecular Compounds. VIII— Copolymerisation of γ-Silicon containing Simple Vinyl Ethers and Methylmethacrylate

A. M. Khomutov, I. A. Shikhiev, N. V. Komarov, and A. P. Alimov

Izvestiya Akad. Nauk. S.S.S.R., otdel. khim. nauk, (1), 140–143 (Jan. 1959)

Conditions of polymerisation of the vinyl ethers of γ-hydroxypropyl-trimethyl- and -methyldiethyl-silanes (I) with methyl methacrylate (II) have been investigated and the corresponding copolymers prepared. Yield and composition depend upon conc. of I and II. Dinitrile of azoisobutyric acid and benzoyl peroxide do notinitia te polymerisation of I. G.J.K.

Heterochain Polyesters

V. V. Korshak and S. V. Vinogradova

XII— Polyesters of Azobenzene-3,3′- and Azobenzene-4,4′-dicarboxylic acids

Izvestiya Akad. Nauk. S.S.S.R., otdel. khim. nauk, (1), 148–153 (Jan. 1959)

The preparation and properties of the polyesters of azobenzene-3,3′- (I) and -4,4′- (II) dicarboxylic acids respectively with aliphatic glycols are described. Polyesters of I yield long coloured fibres, whilst those of II do not form fibres.

XIII— Polyesters of *p*-Xylylene Glycol

Ibid., 154–161

Preparation and properties of the polyesters of *p*-xylylene glycol with 13 aliphatic and 14 aromatic dicarboxylic acids are described. The distribution of fibre-forming and non-fibre-forming polyesters seems to be random.

XIV— Polyesters of *m*-Xylylene Glycol

Ibid., (2), 338–343 (Feb. 1959)

Polyesters of *m*-xylylene glycol with 12 aliphatic and 9 aromatic dicarboxylic acids have been prepared and properties tabulated. All of the polymers form fibres. G.J.K.

PATENTS

Light Coloured Butyl Rubbers

Esso Research & Engineering Co. *USP* 2,873,784

Butyl rubber (100) compounded with CaO (15–80) and MgO (C.I. 77711) (10–50) and, if desired, TiO_2 (C.I. Pigment White 6) (10–150) and ZnO (C.I. Pigment White 4) (5–80), is a particularly good white after vulcanisation. The vulcanised product is self-cleaning and has improved resistance to weathering and discoloration. C.O.C.

Increasing the Opacity of Thermoplastic Resins

Union Carbide Corpn. *BP* 814,635

Addition of 2·5–25% by wt. of TiO_2 (C.I. Pigment White 6) and 0·01–0·3% of aluminium powder or flake (C.I. Pigment Metal I) imparts greatly increased opacity to thermoplastic resin compositions without impairing physical properties or colour. The aluminium must be of such particle size that its covering area on water is 7000–25,000 sq.cm./g. C.O.C.

Water-repellent Finish for Plastics

Bradford Dyers' Assocn. *BP* 814,899

Application to the non-porous surface of a plastic of an organosilicon compound and a compound which hydrolyses to yield titanium oxide or hydroxide, imparts a highly water-repellent finish. C.O.C.

Plastic Sheets Incorporating Dye Images

International Polaroid Corpn. *BP* 814,819

Treatment with an aqueous solution containing iodine ions speeds up the transfer of dye and improves the density of the image of transparent, linear, orientated high mol. wt. hydroxyl containing vinyl polymers which are being printed on by a transfer process using dichroic or isotropic dyes. C.O.C.

Linoleum Composition

Armstrong Cork Co. *USP* 2,873,201

A linoleum composition containing 55–80% by weight of fillers and pigments the remainder being a linoleum cement obtained by heating dimerised fatty acid with pentaerythritol, is readily air-cured at 170–210°F. The product has unusual properties of alkali resistance, indentation, residual indentation, water absorption, stiffness and abrasion resistance. C.O.C.

Titanium Dioxide for Pigmenting Fibres and Films of Synthetic Polymers (IV p. 436)

XIV— ANALYSIS; TESTING; APPARATUS

2 - Methoxy - 6 - chloro - 7 - amino - 9 - (2-diethylamino - 1 - methylbutylamino)acridinedihydrochloride as a Luminescent Acid-Base Indicator

L. P. Zharov and V. L. Zolotavin

Trudy Ural. Politekh. Inst., Sbornik, (57), 76–8 (1956): *Referat Zhur., Met.* Abstr. No. 9260 (1957): *Chem. Abs.*, **53**, 5007 (25 March 1959)

An alcoholic solution of the above indicator fluoresces bright-green under ultraviolet irradiation. Added to an acid solution it causes orange luminescence which becomes green on alkalisation. It is possible to determine the concentration of acids in coloured solutions by using this indicator. A 300 w. mercury lamp was used as the ultraviolet source. The transition interval is within the pH range 6·7–7·3. Index of titration is 7. Relative error is $\not> 0{\cdot}1\%$. C.O.C.

Determination of a Nonionic Detergent when mixed with Soap

C. B. Stuffins

Soap, Perfumery and Cosmetics, **31**, 369–370 (1958)

The nonionic detergent is separated out as a phosphomolybdic complex in presence of dioxane. Thus place a 5 g. sample in a 250 ml. beaker in an oven at 100°C. for 30 min., cool, add 20 ml. dioxane, cover up and leave for 2 hr. Filter through a No. 4 sintered glass crucible, wash out in a beaker with 3 10-ml. portions of dioxane. Transfer the dioxane extract to a 250 ml. beaker, bring to 150 ml. with water, add 5 ml. 20% HCl, 5 ml. 10% $BaCl_2$ and 5 ml. dioxane. Boil and stand for 18 hr. Filter on glass and wash with 100 ml. water. Dry at 100°C. for 2 hr. and weigh. The proportion of detergent in the sample is calculated from a graph prepared from known weights of a particular detergent. C.O.C.

Volumetric Determination of Zinc in Pigments by using an Oxidation-Reduction Indicator

J. Faure *Peintures, Pigments, Vernis*, **33**, 340–2 (1957): *Chem. Abs.*, **53**, 4767 (10 March 1959)

The standard potentiometric method of titrating Zn with $K_4Fe(CN)_6$ is applicable to pigments, which are advantageously dissolved in 25% H_2SO_4. C.O.C.

Comparison of Accelerated and Exterior Weathering of Paints

W. Funke, D. Walper, and R. Haug

Deut. Farben-Z., **12**, 478–484 (1958): *Chem. Abs.*, **53**, 5699 (25 March 1959)

The main factors responsible for the weathering of paints were correlated in pairs: water/light (WL), water/heat (WH), cold/heat (CH), water/cold (WC), 16 8-hr. cycles being used. The paints were immersed in water to obtain uniform swelling. The following types of paint were studied: stand oil-ZnO, alkyd-ZnO, epoxy-TiO_2, urethane-TiO_2 and polyester-TiO_2, changes in penetration and pendulum hardness, Philips indentation, and gloss being recorded together with exposure time. Gloss is most affected by the WL cycle, the WH and CH cycles affect the whole film making it harder and less elastic. The WL cycles corresponded best with atmospheric weathering partly because the ultraviolet radiation used also heated the paints to 50–60°C. A combination of 15–25 hr. immersion, 4 hr. light and a short heat period is recommended as an accelerated weathering test. Daylight lamps are prepared for testing pigments. Constant time factors to convert accelerated test results to outdoor durability are not feasible. Film properties change at varying rates with different paints. In addition variations in annual and

seasonal weather conditions make reproducibility and duplication impossible. Differences between binder types and relative ratings within each type obtained by accelerated weathering tests generally agree with the results of outdoor exposure. C.O.C.

Separation and Analysis of Scale-rich Material from Wool
J. H. Bradbury *Nature*, **183**, 305–306 (31 Jan. 1959)

Wool scales isolated by chemical and enzymatic methods are degraded to an unknown extent. This difficulty is overcome in the present abrasion technique. The descaler consists of a chisel-edged glass rod which has a groove along the edge. When a single fibre is passed through the groove, the scales are dislodged and collect on the sides of the descaler. The product contains rather more of the wool substance than the scales alone, the yield is 0·5 mg. from 200–400 fibres. Compared with fibre composition, the scales contain more proline and less aspartic and glutamic acids. There are also differences in the composition of scales from different wools. E.V.T.

Relationship between the Affinity of Direct Dyes and their R_f Values
T. Urahata *Kôgyo Kagaku Zasshi*, **60**, 292–4 (1957): *Chem. Abs.*, **53**, 5683 (25 March 1959)

Various direct dyes were dissolved in 15 vol. % pyridine (5×10^{-4} mole/l.) and subjected to paper chromatography at 20°c. Dyes of lower R_f values have higher affinity for cotton at 90°c. The affinity value is proportional to $\log (1-R_f)/R_f$. C.O.C.

Measurement of the Resolving Power and Colour Rendering of Colour Films
S. Rösch
Wiss. Phot., Intern. Konf. Köln, 658–666 (1956)

Gloss Measurement
H. Haussühl and K. Hamann
Farbe u. Lack, **64**, 642–9 (1958): *Chem. Abs.*, **53**, 5699 (25 March 1959)

Total or diffuse and specular or mirror reflection are different kinds of gloss. Gloss meters do not measure both kinds and sometimes disagree with visual gloss ratings of paints. A gloss meter has been developed which measures with a moving photoelectric cell the intensity of a fixed incident beam of light dispersed on the surface in all directions. The intensities plotted against the corresponding emergent angles give a curve with a steep maximum at the regular reflectance angle. The rest of the curve slowly flattens out with increasing dispersion angle and represents background reflection from the pigment layer. The gloss is characterised by the maximum intensity, *Ir*, and the width of the curve at half the maximum intensity value, *Ho*. On a gloss diagram constructed from these two values mirror surfaces with high *Ir* appear near the top left while matte surfaces with high *Ho* are toward the right. Curves of equal visual gloss show that at lower reflection *Ho* is less important for gloss ratings, while at high reflectance the half-value decreases with increasing sharpness of image. The gloss measurements of isochromatic paints agree with visual ratings. Surface structure is also determined by interference line microscope pictures, which demonstrate microscopic roughness and optical flatness. The methods are used to measure weathering effects on coated surfaces quicker and more accurately. C.O.C.

Silica Determinations in White Rubber Fillers
M. Ceamiş and A. Kiriţesca
Ind. uşoară (Bucharest), **3**, 201–2 (1956)

A rapid method to distinguish between colloidal silica and alumina, kaolin or other fillers used for white rubber is to heat a sample with a few drops of $HClO_4$, then with $KHSO_4$ to 600°c., where the SO_3 formed reacts with the metasilicates to form SiO_2 and H_2SiO_3. After heating to 900°c., the crucible is quenched in cold water and the melt extracted with HCl. After boiling it, the extract is filtered and the residue calcined and weighed. C.O.C.

PATENTS

Measuring the Moisture Content of Material of High Resistivity
Isotope Products *BP* 814,395

Apparatus comprising a set of electrodes which make electrical contact with the material to be tested, e.g. a moving web of paper. They are supplied with a.c. current. Drop in the voltage between the electrodes operates a signal voltage which is amplified. The resistive components of the current flow are filtered out and measured. The apparatus has only a very slight possible source of error. C.O.C.

Determining and Controlling the Moisture Content of Dielectric Materials
Commonwealth Scientific & Industrial Research Organisation *BP* 815,000

The material is placed between the plates of a test condenser into which at least two voltages of different frequencies are simultaneously fed. This yields a voltage which is a function of the ratio of change in the voltage drops across the plates of the test condenser corresponding to each feed frequency. From this the amount of moisture in the material can be determined. C.O.C.

Formation of Complexes during Periodate Oxidation of Polyols (XI p. 441)

XV— MISCELLANEOUS

Tablet Colour Coating with Pigments
S. J. Tucker, A. E. Nicholson, and H. Engelbert
J. Am. Pharm. Assoc., **47**, 849–850 (1958): *Chem. Abs.*, **53**, 4657 (10 March 1959)

Insoluble pigments dispersed in TiO_2 syrup have the following advantages over water-soluble dyes for colour-coating of tablets: immediate application following the rounding-coat thus eliminating the smoothing coats, reduction in time and number of coats, easy matching from lot to lot, and prevention of overcolouring. C.O.C.

Patenting of Chemical Inventions
J. M. Soesan
Chem. and Ind., (50), 1640–1645 (13 Dec. 1958)

A discussion of the main requirements for patentability and the difficulties which may arise in the patenting of chemical inventions. A number of examples of patent litigation are quoted. A.J.

PATENT

Fluorescent Resin Compositions as Marking Agents
Hodogaya Chemical Co. *BP* 814,628

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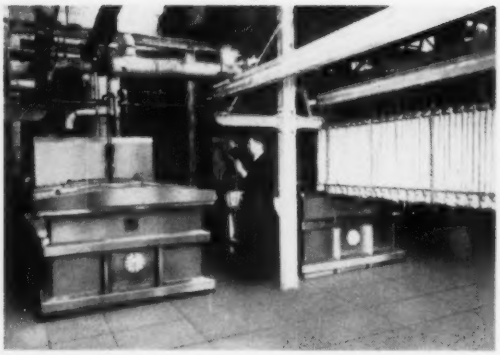

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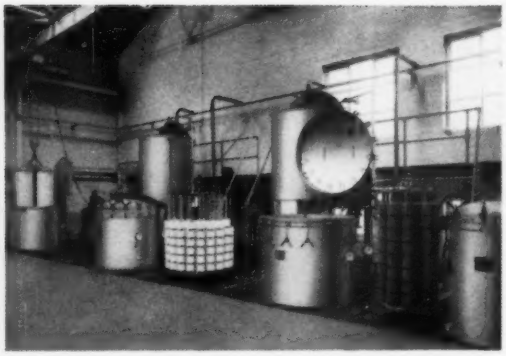

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APPOINTMENTS etc

Advertisements relating to APPOINTMENTS VACANT, APPOINTMENTS WANTED, and MISCELLANEOUS ITEMS are invited for insertion on this page. Advertisements of Appointments Wanted are gratis to members, but must not exceed twenty-four words.

All inquiries relating to Advertisements should be addressed to THE GENERAL SECRETARY, THE SOCIETY of DYERS and COLOURISTS, DEAN HOUSE, 19 PICCADILLY, BRADFORD 1, YORKSHIRE.

Replies may be addressed BOX —, THE SOCIETY of DYERS and COLOURISTS, DEAN HOUSE, 19 PICCADILLY, BRADFORD 1, YORKSHIRE, where all communications relating to these Advertisements are treated in strict confidence.

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DYEING AND FINISHING

APPLICATIONS are invited for several vacancies caused by the expansion of the Textile Development Department of BRITISH NYLON SPINNERS. Candidates should have a degree in chemistry or textile chemistry, of at least 2nd Class Honours standard, and some experience in finishing methods is desirable; for one of the appointments specialist experience in proofing techniques would be most valuable. The work may be of two kinds: (i) Technical service to the trade either as a Representative or in the laboratory. (ii) Development work primarily in the laboratory on new methods of finishing nylon fabrics.

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APPLICATIONS are invited for the position of SENIOR LECTURER in TEXTILE CHEMISTRY in the College of Technology, Belfast.

This position involves responsibility for a university degree course of The Queen's University of Belfast. Good facilities are available for research work, which will be encouraged.

The salary is in accordance with the salary scales of the Ministry of Education for Northern Ireland, which are at present under review. £1,417 10s 0d rising to £1,627 10s 0d per annum.

Further particulars and forms of application may be obtained from the Principal, College of Technology, Belfast, with whom completed forms should be lodged not later than FRIDAY, 2nd October 1959.

Canvassing is strictly forbidden and will disqualify.

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ASSISTANT DYER wanted for piece goods dyers in the London area, equipped with jigs, winches, etc., and handling vats, directs, cross-dyes, azoics, etc. Some practical experience desirable, but sound basic knowledge essential. This is an excellent opening for a young man with an eye to the future. Write in the first instance, stating age, qualifications, present salary and full details of training and experience. All applications will be treated with strict confidence. BOX V180

CHEMIST required with sound training in dyestuff technology preferably with some experience in colour matching for Rayon and Synthetic fibres, by Piece Goods Dyers in North West Midlands. BOX V175

DYEHOUSE Manager required for well known firm in Manchester area specialising in yarn processing, particularly vat dyeing of cotton hanks. Applicants must have practical knowledge of fast dyeing processes and working to a high standard of matching. State age, experience and salary required. BOX V174

EXPERIENCED Dyer required by Leicester hank yarn dyeing firm. Applicant must be conversant with the application of all classes of fast dyes to wool, preferably with some experience of other fibres. Willing to supervise evening shifts. Salary £900–£1,500, according to ability. BOX V177

FOREMAN Dyer required for Cotton Dyehouse situated in South Wales. To be a good shade matcher and able to control labour. BOX V179

HARDMAN & HOLDEN LTD. require a qualified chemist for research work connected with the application of chemicals to problems in the Textile Industry. The work is varied and interesting, and will involve some customer contacts. Applicants should preferably be Organic Chemists with some experience of textile chemistry.

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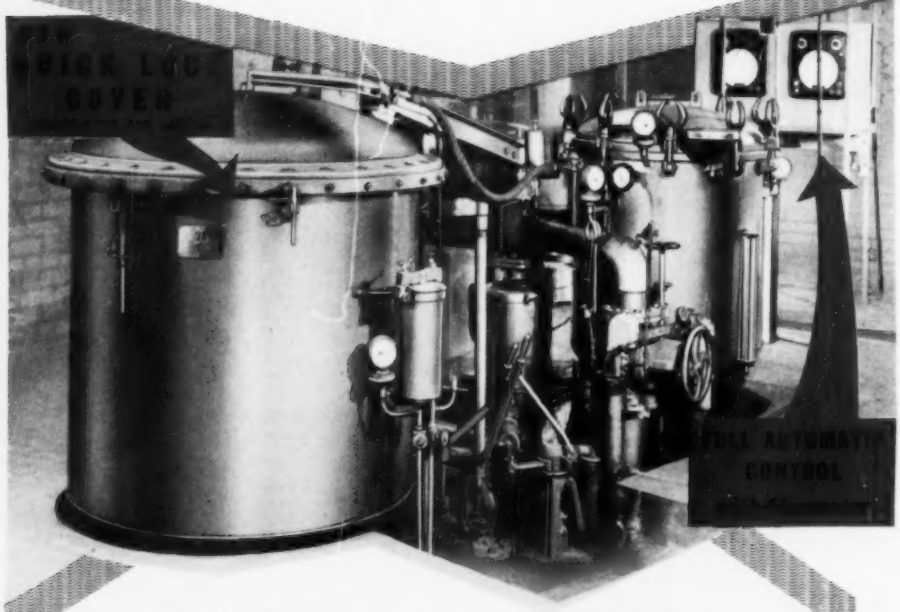

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